Remedial Action Contract for Remedial Response, Enforcement Oversight, and Non-Time Critical Removal Activities at Sites of Release or Threatened Release of Hazardous Substances in EPA Region 8

U.S. EPA Contract No. EP-W-05-049

Sampling and Analysis Plan/Quality Assurance Project Plan Flowerbed Sampling Study Libby Asbestos Site, Operable Unit 4

Revision 0 - August 2012

Work Assignment No.: 329-RICO-08BC

Libby Asbestos Superfund Project,

OU4 Remedial

Investigation/Feasibility Study

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A PROJECT MANAGEMENT

A1. Title and Approval Page

Title:

Sampling and Analysis Plan/Quality Assurance Project Plan, Flowerbed Sampling Study Libby Asbestos Site, Operable Unit 4, Revision 0 - August 2012

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Sampling and Analysis Plan/Quality Assurance Project Plan Flowerbed Sampling Study Libby Asbestos Site, Operable Unit 4

REVISION LOG:

Revision No.	Date	Description
0	8/10/12	

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List of Acronyms and Abbreviations

% percent > greater than

greater than or equal to ≥ ≤ less than or equal to

Ago grid opening area

cubic centimeter CC

CDM Smith CDM Federal Programs Corporation

CERCLA Comprehensive Environmental Response, Compensation, and Liability Act

CHISQ chi-squared

CI confidence interval cm⁻² per square centimeter COC chain-of-custody record

DQO data quality objective

ED exposure duration

EDD electronic data deliverable EDS energy dispersive spectroscopy

energy dispersive x-ray analysis EF exposure frequency

EDXA

EFA effective filter area **EPA** U.S. Environmental Protection Agency

ERT Environmental Response Team

ESAT Environmental Services Assistance Team

EΤ exposure time

f indirect preparation dilution factor

f/cc fibers per cubic centimeter

FBAS fluidized bed asbestos segregator

FSDS field sample data sheet

ft2 square foot FTL field team leader

gram g-1 per gram

GIS geographic information system GO_x number of grid openings GPI general property investigation **GPS** global positioning system

HASP Health and Safety Plan

HAZWOPER Hazardous Waste Operations and Emergency Response

H&S Health and Safety

HDPE high density polyethylene

ID identification IUR inhalation unit risk

IDW investigation-derived waste

IRIS Integrated Risk Information System

L liters

L/cc liters per cubic centimeter

L/min liters per minute
LA Libby amphibole

LADT Libby Asbestos Data Tool LC laboratory coordinator

MDEQ Montana Department of Environmental Quality

mm millimeter

mm² square millimeter

N number

N/A not applicable

NIST National Institute of Standards and Technology

NVLAP National Voluntary Laboratory Accreditation Program

OSHA Occupational Safety and Health Administration

PCM phase contrast microscopy

PCME phase contrast microscopy-equivalent

PE performance evaluation PLM polarized light microscopy

PLM-VE polarized light microscopy visual area estimation

QA quality assurance

QAM quality assurance manager
QAPP quality assurance project plan
QA/QC quality assurance/quality control
QATS Quality Assurance Technical Support

QC quality control

RBC risk-based concentration

RPM Regional Project Manager ROM Record of Modification

s/cm² structures per square centimeter

s/g structures per gram

SAP sampling and analysis plan SAED selective area electron diffraction

Shaw Shaw Environmental, Inc.
Site Libby Asbestos Superfund Site
SOP standard operating procedure
SPF Sample Preparation Facility
SRM standard reference materials

TAS target analytical sensitivity

TEM transmission electron microscopy

short-term exposure limit

TWA time-weighted average TWF time-weighting factor

STEL

USGS United States Geological Survey

V air sample volume

VWC volumetric water content

wt% mass percent

μm micrometers

A3. Distribution List

Copies of this completed and signed sampling and analysis plan/quality assurance project plan (SAP/QAPP) should be distributed to:

U.S. Environmental Protection Agency, Region 8

1595 Wynkoop Street

Denver, Colorado 80202-1129

- Victor Ketellapper, Ketellaper. Victor@epa.gov (1 hard copy, electronic copy)
- Elizabeth Fagen, <u>Fagen.Elizabeth@epa.gov</u> (electronic copy)
- Don Goodrich, <u>Goodrich.Donald@epa.gov</u> (electronic copy)
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- Dania Zinner, Zinner. Dania@epa.gov (electronic copy)
- David Berry, <u>Berry.David@epa.gov</u> (electronic copy)

EPA Information Center - Libby

108 East 9th Street

Libby, Montana 59923

- Mike Cirian, <u>Cirian.Mike@epa.gov</u> (1 hard copy, electronic copy)

Montana Department of Environmental Quality

1100 North Last Chance Gulch

Helena, Montana 59601

- Carolyn Rutland, <u>CRutland@mt.gov</u> (electronic copy)
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Libby, Montana 59923

- Kara McKenzie, <u>mckenzieKE@cdmsmith.com</u> (3 hard copies, electronic copy)
- Terry Crowell, crowellTL@cdmsmith.com (electronic copy)
- Damon Repine, <u>repineDL@cdmsmith.com</u> (electronic copy)

CDM Smith - Denver Office

555 17th Street, Suite 1100

Denver, Colorado 80202

- Nathan Smith, <u>smithNT@cdmsmith.com</u> (electronic copy)

Copies of the SAP/QAPP will be distributed to the individuals above by CDM Federal Programs Corporation (CDM Smith), either in hard copy or in electronic format (as indicated

above). The CDM Smith Project Manager (or their designate) will distribute updated copies each time a SAP/QAPP revision occurs. An electronic copy of the final, signed SAP/QAPP (and any subsequent revisions) will also be posted to the Libby Field eRoom.

A4. Project Task Organization

Figure A-1 presents an organizational chart that shows lines of authority and reporting responsibilities for this project. The following sections summarize the entities and individuals that will be responsible for providing project management, technical support, and quality assurance for this project.

A4.1 Project Management

The U.S. Environmental Protection Agency (EPA) is the lead regulatory agency for Superfund activities within the Libby Asbestos Superfund Site (Site). The EPA Region 8 Libby Asbestos Project Team Leader is Victor Ketellapper. The EPA Regional Project Manager (RPM) for this sampling effort is Elizabeth Fagen. The EPA Region 8 Onsite Field Team Leader for this sampling effort is Michael Cirian.

The Montana Department of Environmental Quality (MDEQ) is the support regulatory agency for Superfund activities at the Site. The MDEQ Project Manager for this sampling effort is Carolyn Rutland. The EPA will consult with MDEQ as provided for by the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the National Contingency Plan, and applicable guidance in conducting Superfund activities.

A4.2 Technical Support

A4.2.1 SAP/QAPP Development

This SAP/QAPP was developed by CDM Smith at the direction of, and with oversight by, the EPA. This SAP/QAPP contains all the elements required for both a SAP and a QAPP and has been developed in general accordance with the EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5 (EPA 2001) and the Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G4 (EPA 2006).

Copies of the SAP/QAPP will be distributed by the CDM Smith Project Manager (or their designate), either in hard copy or in electronic format, as indicated in Section A3. The CDM Smith Project Manager (or their designate) will distribute updated copies each time a SAP/QAPP revision occurs. An electronic copy of the final, signed SAP/QAPP (and any subsequent revisions) will also be posted to the Libby Field eRoom.

A4.2.2 Field Sampling Activities

CDM Smith will also be responsible for conducting all field sampling activities in support of the sampling program described in this SAP/QAPP. Key CDM Smith personnel that will be involved in this sampling program include:

- Nathan Smith, Project Manager
- Kara McKenzie, Field Team Leader
- Tracy Dodge, Sample Coordinator
- Scott Miller, Field Data Manager
- Terry Crowell, Quality Assurance Manager
- Damon Repine, Health and Safety Manager

A4.2.3 Asbestos Analysis

All samples collected as part of this project will be sent for preparation and analysis for asbestos at laboratories selected and approved by the EPA to support the Site. The EPA Environmental Services Assistance Team (ESAT) is responsible for procuring all analytical and preparation laboratory services and providing direction to the analytical laboratories. Don Goodrich (EPA Region 8) is responsible for managing the ESAT laboratory support contract for asbestos. The ESAT Region 8 Team Manager at TechLaw, Inc. is Mark McDaniel. He is also the designated laboratory coordinator (LC) for the Libby project that is responsible for directing the analytical laboratories, prioritizing analysis needs, and managing laboratory capacity.

A4.2.4 Data Management

All data generated as part of this sampling effort will be managed and maintained in Scribe. The EPA Environmental Response Team (ERT) is responsible for the administration of all Scribe data management aspects of this project. Joseph Schafer is responsible for overseeing the ERT data management support contract. ERT is responsible for the development and management of Scribe and the project-specific data reporting requirements for the Libby project.

The CDM Smith field data manager (Scott Miller) is responsible for uploading sample information to the field Scribe project database. ESAT is responsible for uploading new analytical results to the analytical Scribe project database. The ESAT project data manager for the Libby project is Janelle Lohman (TechLaw, Inc.).

Because of the quantity and complexity of the data collected at the Site, the EPA has designated a Libby Data Manager to manage and oversee the various data support contractors. The EPA Region 8 Data Manager for the Libby project is Jeff Mosal.

A4.3 Quality Assurance

There is no individual designated as the EPA Quality Assurance Manager for the Libby project. Rather, the Region 8 QA program has delegated authority to the EPA RPMs. This means that the EPA RPMs have the ability to review and approve governing investigation documents developed by Site contractors. Thus, it is the responsibility of the EPA RPM for this sampling effort (Elizabeth Fagen), who is independent of the entities planning and obtaining the data, to ensure that this SAP/QAPP has been prepared in accordance with the EPA QA guidelines and requirements. The EPA RPM is also responsible for managing and overseeing all aspects of the quality assurance/quality control (QA/QC) program for this sampling effort. In this regard, the RPM is supported by the EPA Quality Assurance Technical Support (QATS) contractor, Shaw Environmental, Inc. (Shaw). The QATS contractor will evaluate and monitor laboratory QA/QC and is responsible for performing annual audits of each analytical laboratory.

Terry Crowell (CDM Smith) is the field Quality Assurance Manager for this project. Ms. Crowell is responsible for evaluating and monitoring field QA/QC, for providing oversight of field sampling and data collection activities, and for designating a qualified individual to conduct the field surveillance (see Section B5.1).

A5. Problem Definition/Background

A5.1 Site Background

Libby is a community in northwestern Montana located 7 miles southwest of a vermiculite mine that operated from the 1920s until 1990. The mine began limited operations in the 1920s and was operated on a larger scale by the W.R. Grace Company from approximately 1963 to 1990. Studies revealed that the vermiculite from the mine contains amphibole-type asbestos, referred to as Libby amphibole (LA).

Epidemiological studies revealed that workers at the mine had an increased risk of developing asbestos-related lung disease (McDonald *et al.* 1986a,b, Amandus and Wheeler 1987, Amandus *et al.* 1987, Sullivan 2007). Additionally, radiographic abnormalities were observed in 17.8 percent of the general population of Libby including former workers, family members of workers, and individuals with no specific pathway of exposure (Peipins *et al.* 2003). Although the mine has ceased operations, historic or continuing releases of LA from mine-related materials could be serving as a source of on-going exposure and risk to current and future residents and workers in the area. The Site was listed on the National Priorities List in October 2002.

A5.2 Reasons for this Project

Previous investigations conducted at residential properties at the Site have shown that soils in flowerbeds frequently contain visible vermiculite. When visible vermiculite is noted in flowerbeds during the general property investigation (GPI), soil samples are not collected from the flowerbed for asbestos analysis. This is because the presence of visible vermiculite in flowerbeds (which is considered a "specific-use area" or SUA) is a primary trigger for performing a soil removal at the property (EPA 2003). However, some property owners have indicated that, in cases where visible vermiculite is present in the flowerbed, the soils in the flowerbed were derived from store-bought potting soil, which are not expected to contain LA. This suggests that the identification of visible vermiculite in a flowerbed may not be a reliable indicator of mine contamination (i.e., LA), and that some soil removals could be performed unnecessarily as a consequence of this trigger. Therefore, the purpose of this study is to collect data that can be used to evaluate if the presence of visible vermiculite in flowerbeds is a reliable indicator of mine contamination at a property. This study also seeks to collect data on whether other information gained about the property (e.g., year the property was built, homeowner input on flowerbed soil source) can be used to guide decision-making on soil removals for flowerbeds.

A5.3 Applicable Criteria and Action Limits

At the Libby Site, the EPA has developed action levels and removal criteria for LA that are applicable to emergency response actions performed at residential/commercial properties (EPA 2003). In accordance with the action memo, the presence of visible vermiculite in an SUA, such as a flowerbed, garden, play area, or unpaved driveway, is a primary trigger for performing a soil removal at the property. Once a removal is triggered for a property, soil removal is performed for all non-SUA areas (e.g., yards), excluding crawlspaces and basements, where measured soil concentrations of LA are detect by polarized light microscopy using visual area estimation (PLM-VE) (i.e., results are reported as Bin B1, B2, or C), or where visible vermiculite is observed. An exception to this rule is made and removal is not performed for limited use areas (LUAs) where soil concentration is trace for LA and visible vermiculite observations are low.

Currently, there are no established criteria to directly evaluate human health risk based on levels of LA in soil.

A6. Project/Task Description

A6.1 Task Summary

As noted above, the purpose of this study is to evaluate if the presence of visible vermiculite in flowerbeds is a reliable indicator of mine contamination at a property. This study will include four tasks designed to address this question.

Task 1 - Flowerbeds in Libby

In Task 1, soil samples will be collected from flowerbeds at residential properties within Libby where visible vermiculite is present. These soil samples will be analyzed for LA by transmission electron microscopy (TEM) after preparation by a fluidized bed asbestos segregator (FBAS)¹ and by the Libby-specific PLM methods.

Task 2 - Flowerbeds in Troy

In Task 2, previously collected soil samples from flowerbeds at residential properties in Troy where visible vermiculite is present will be pulled from the soil archive and re-analyzed for asbestos using FBAS-TEM and the Libby-specific PLM methods.

Task 3 - Store-bought Materials from Libby

In Task 3, previously collected soil samples of store-bought potting soil purchased in Libby will be pulled from the soil archive and re-analyzed for asbestos using FBAS-TEM and the Libby-specific PLM methods. This task will also include the collection of samples from store-bought bagged vermiculite purchased from retail stores in Libby for analysis of asbestos using FBAS-TEM and the Libby-specific PLM methods.

Task 4 - Store-bought Materials from Other Cities

In Task 4, samples will be collected of store-bought potting soil purchased from retail stores outside of Libby (e.g., Denver) and analyzed for asbestos using FBAS-TEM and the Libby-specific PLM methods.

A6.2 Work Schedule

All sample collection and archive sample retrieval will be performed in the summer of 2012. Sample analysis and data evaluation and interpretation tasks will be performed over the summer and fall of 2012.

A6.3 Locations to be Evaluated

Section B1.1 provides detailed information on the locations that will be sampled as part of Tasks 1 and 4, as well as the sample selection criteria that will be used for Tasks 2 and 3.

¹ For the purposes of this SAP/QAPP, this soil preparation and analysis method is referred to as FBAS-TEM.

A6.4 Resources and Time Constraints

As noted above, the sampling for Task 1 and Task 4 is scheduled to occur in the summer of 2012. The EPA has both resource and time constraints with the scope of this sampling program. This sampling program will be limited to approximately ten samples for Task 1, twelve samples for Task 2, six samples for Task 3 (3 potting soil, 3 bagged vermiculite), and six samples for Task 4. Sample collection and analysis needs to be performed quickly, as there are several properties where soil removals have been placed "on hold" pending the outcome of this study.

A7. Quality Objectives and Criteria

A7.1 Data Quality Objectives

Data quality objectives (DQOs) are statements that define the type, quality, quantity, purpose, and use of data to be collected. The design of a study is closely tied to the DQOs, which serve as the basis for important decisions regarding key design features such as the number and location of samples to be collected and types of analyses to be performed. The EPA has developed a seven-step process for establishing DQOs to help ensure that data collected during a field sampling program will be adequate to support reliable site-specific decision-making (EPA 2001, 2006).

Appendix A provides the detailed implementation of the seven-step DQO process associated with this SAP/QAPP.

A7.2 Performance Criteria

The range of LA concentrations that will be measured in flowerbeds and store-bought materials by FBAS-TEM is not known. However, based on the available PLM-VE results for these types of samples that are available, which tended to be Bin A (non-detect) and Bin B1 (trace), it is likely that concentrations will be low (<0.2 percent [%]). Thus, the analytical requirements for LA measurements by FBAS-TEM, as established in Section B4, ensure concentrations will be reliably detected and quantified if present at levels similar to those measured in other low levels studies (e.g., borrow source and background areas) and will be comparable to other FBAS-TEM results from other sampling efforts.

A7.3 Precision

The precision of asbestos measurements for TEM is determined mainly by the number (N) of asbestos structures counted in each sample. The coefficient of variation resulting from random Poisson counting error is equal to $1/N^{0.5}$. In general, when good precision is needed, it is

desirable to count a minimum of 3-10 structures per sample, with counts of 20-25 structures per sample being optimal.

A7.4 Bias/Accuracy and Representativeness

To the extent feasible, samples should be collected and analyzed in accordance with procedures that have been performed in previous (and planned future) soil sampling efforts. This will ensure that the results of this study are representative and appropriate for comparison to other data sets.

A7.5 Completeness

Target completeness for this project is 100%. If any samples are not collected, or if the TEM analysis is not completed successfully, this could result in that portion of the study providing no useful information. In this event, additional sampling may be needed to support EPA decision-making.

A7.6 Comparability

The data generated during this study will be obtained using standard analytical methods for LA that have been utilized previously in other studies, and will yield data that are comparable to previous analyses of LA in soil.

A7.7 Method Sensitivity

The method sensitivity (analytical sensitivity) needed for LA analysis is discussed in Section B4.

A8. Special Training/Certifications

A8.1 Field

Asbestos is a hazardous substance that can increase the risk of cancer and serious non-cancer effects in people who are exposed by inhalation. Therefore, all individuals involved in the collection, packaging, and shipment of samples must have appropriate training. Prior to starting any field work, any new field team member must complete the following, at a minimum:

Training Requirement	Location of Documentation Specifying Training Requirement Completion
Read and understand the governing Health and Safety Plan (HASP)	HASP signature sheet
Attend an orientation session with the field health and safety (H&S) manager	Orientation session attendance sheet

Training Requirement	Location of Documentation Specifying Training Requirement Completion
Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operations and Emergency Response (HAZWOPER) and relevant 8-hour refreshers	OSHA training certificates
Current 40-hour HAZWOPER medical clearance	Physician letter in the field personnel files
Respiratory protection training, as required by 29 CFR 1910.134	Training certificate
Asbestos awareness training, as required by 29 CFR 1910.1001	Training certificate
Sample collection techniques	Orientation session attendance sheet

All training documentation will be stored in the CDM Smith field office. It is the responsibility of the field H&S manager to ensure that all training documentation is up-to-date and on-file for each field team member.

Prior to beginning field sampling activities, a field planning meeting will be conducted to discuss and clarify the following:

- Objectives and scope of the fieldwork
- Equipment and training needs
- Field operating procedures, schedules of events, and individual assignments
- Required quality control (QC) measures
- Health and safety requirements

It is the responsibility of each field team member to review and understand all applicable governing documents associated with this sampling program, including this SAP/QAPP, all associated standard operating procedures (SOPs) (see **Appendix B**), and the applicable HASP.

A8.2 Laboratory

A8.2.1 Certifications

All analytical laboratories participating in the analysis of samples for the Libby project are subject to national, local, and project-specific certifications and requirements. Each laboratory is accredited by the National Institute of Standards and Technology (NIST)/National Voluntary Laboratory Accreditation Program (NVLAP) for the analysis of airborne asbestos by transmission electron microscope (TEM) and/or analysis of bulk asbestos by polarized light microscopy (PLM). This includes the analysis of NIST/NVLAP standard reference materials (SRMs), or other verified quantitative standards, and successful participation in two proficiency rounds per year each of bulk asbestos by PLM and airborne asbestos by TEM supplied by NIST/NVLAP.

Copies of recent proficiency examinations from NVLAP or an equivalent program are maintained by each participating analytical laboratory. Many of the laboratories also maintain certifications from other state and local agencies. Copies of all proficiency examinations and certifications are also maintained by the LC.

Each laboratory working on the Libby project is also required to pass an on-site EPA laboratory audit. The details of this EPA audit are discussed in Section B5.3.3. The LC also reserves the right to conduct any additional investigations deemed necessary to determine the ability of each laboratory to perform the work. Each laboratory also maintains appropriate certifications from the state and possibly other certifying bodies for methods and parameters that may also be of interest to the Libby project. These certifications require that each laboratory has all applicable state licenses and employs only qualified personnel. Laboratory personnel working on the Libby project are reviewed for requisite experience and technical competence to perform asbestos analyses. Copies of personnel resumes are maintained for each participating laboratory by the LC in the Libby project file.

A8.2.2 Laboratory Team Training/Mentoring Program

Initial Mentoring

The orientation program to help new laboratories gain the skills needed to perform reliable analyses at the Site involves successful completion of a training/mentoring program that was developed for new laboratories prior to their analysis of Libby field samples. All new laboratories are required to participate in this program. The training program includes a rigorous 2-3 day period of on-site training provided by senior personnel from those laboratories already under contract on the Libby project, with oversight by the QATS contractor. The tutorial process includes a review of morphological, optical, chemical, and electron diffraction characteristics of LA, as well as training on project-specific analytical methodology, documentation, and administrative procedures used on the Libby site. The mentor will also review the analysis of at least one sample by each type of analytical method with the trainee laboratory.

Site-Specific Reference Materials

Because LA is not a common form of asbestos, U.S. Geological Survey (USGS) prepared Site-specific reference materials using LA collected at the Libby mine site (EPA 2008a). Upon entry into the Libby program, each laboratory is provided samples of these LA reference materials. Each laboratory is required to analyze multiple LA structures present in these samples by TEM in order to become familiar with the physical and chemical appearance of LA and to establish a reference library of LA energy dispersive spectroscopy (EDS) spectra. These laboratory-specific and instrument-specific LA reference spectra (EPA 2008b) serve to guide the classification of asbestos structures observed in Libby field samples during TEM analysis.

Regular Technical Discussions

On-going training and communication is an essential component of QA for the Libby project. To ensure that all laboratories are aware of any technical or procedural issues that may arise, a regular teleconference is held between the EPA, their contractors, and each of the participating laboratories. Other experts (e.g., USGS) are invited to participate when needed. These calls cover all aspects of the analytical process, including sample flow, information processing, technical issues, analytical method procedures and development, documentation issues, project-specific laboratory modifications, and pertinent asbestos publications.

Professional/Technical Meetings

Another important aspect of laboratory team training has been the participation in technical conferences. The first of these technical conferences was hosted by USGS in Denver, Colorado, in February 2001, and was followed by another held in December 2002. The Libby laboratory team has also convened on multiple occasions at the ASTM Johnston Conference in Burlington, Vermont, including in July 2002, July 2005, July 2008, and July 2011, and at the Michael E. Beard Asbestos Conference in San Antonio, Texas in January 2010. In addition, members of the Libby laboratory team attended an EPA workshop to develop a method to determine whether LA is present in a sample of vermiculite attic insulation held in February 2004 in Alexandria, Virginia. These conferences enable the Libby laboratory and technical team members to have an on-going exchange of information regarding all analytical and technical aspects of the project, including the benefits of learning about developments by others.

A8.2.3 Analyst Training

All TEM analysts for the Libby project undergo extensive training to understand TEM theory and the application of standard laboratory procedures and methodologies. The training is typically performed by a combination of personnel, including the laboratory manager, the laboratory quality assurance manager (QAM), and senior TEM analysts.

In addition to the standard TEM training requirements, trainees involved with the Libby project must familiarize themselves with Site-specific method deviations, project-specific documents, and visual references. Standard samples that are often used during TEM training include known pure (traceable) samples of chrysotile, amosite, crocidolite, tremolite, actinolite and anthophyllite, as well as fibrous non-asbestos minerals such as vermiculite, gypsum, antigorite, kaolinite, and sepiolite. New TEM analysts on the Libby project are also required to perform an EDS spectra characterization evaluation on the LA-specific reference materials (similar to EPA 2008b) provided during the initial training program to aide in LA mineralogy recognition and definition. Satisfactory completion of each of these tasks must be approved by a senior TEM analyst.

All TEM analysts are also trained in the Site-specific laboratory QA/QC program requirements for TEM (see Section B5.3.4). The entire program is discussed to ensure understanding of requirements and responsibilities. In addition, analysts are trained in the project-specific reporting requirements and data reporting tools utilized in transmitting results. Upon completion of training, the TEM analyst is enrolled as an active participant in the Libby laboratory program.

A training checklist or logbook is used to assure that the analyst has satisfactorily completed each specific training requirement. It is the responsibility of the laboratory QAM to ensure that all TEM analysts have completed the required training requirements.

A9. Documentation and Records

A9.1 Field

Field teams will record sample information on the most current version of the Site-specific field sample data sheets (FSDSs) developed for each medium². Section B3.1.2 provides detailed information on the documentation requirements for FSDS forms. In brief, the FSDS forms document the unique sample identifier assigned to every sample collected as part of this program. In addition, the FSDSs provide information on whether the sample is representative of a field-based QC sample (e.g., field blank, field duplicate).

A9.2 Laboratory

All preparation and analytical data for asbestos generated in the laboratory will be documented on Site-specific laboratory bench sheets and entered into a database or spreadsheet electronic data deliverable (EDD) for submittal to the data managers. Section B4.2 provides detailed information on the requirements for laboratory documentation and records.

A9.3 Logbooks and Records of Modification/Deviations

It is the also responsibility of the field team, preparation laboratory, and analytical laboratory staff to maintain logbooks and other internal records throughout the sample lifespan as a record of sample handling procedures. Significant deviations (i.e., those that impact or have the potential to impact investigation objectives) from this SAP/QAPP, or any procedures referenced herein governing sample handling, will be discussed with the EPA Project Manager (or their designate) and the CDM Smith Project Manager prior to implementation. Such deviations will be recorded on a Record of Modification (ROM) form. Sections B5.1.2, B5.2.2, and B5.3.2 provide detailed information on the procedures for preparing and submitting ROMs by field, preparation laboratory, and analytical laboratory personnel, respectively.

² The most recent versions of the FSDS forms are provided in the Libby Field eRoom.

B DATA GENERATION AND ACQUISITION

B1. Study Design

This study seeks to collect data to determine if the presence of visible vermiculite in a flowerbed is a reliable indicator of mine contamination at a property. As described in Section A6.1, this study has been split into four tasks. The study design for each task is described below.

B1.1 Description by Task

B1.1.1 Task 1 - Flowerbeds in Libby

Property selection criteria for Task 1 were developed to target properties in Libby that have flowerbeds with visible vermiculite. Additionally, these criteria are designed to evaluate if information on the year that the property was built can be used to guide decisions on whether the presence of visible vermiculite in a flowerbed is an indicator of mine contamination. Two different types of properties will be sampled as part of this task – properties built in or prior to 1995 (Category 1) and properties built after 1995 (Category 2). The cut-off date of 1995 was selected because mine operations ceased in 1990; thus, a five-year buffer is provided between when mine operations ceased and when it is likely that mine material was no longer being actively used in flowerbeds. For both categories of property, the following criteria apply:

- Property has had a detailed investigation portion of a GPI (i.e., a primary removal trigger was found in an SUA)
- Visible vermiculite was observed in a flowerbed
- Property has NOT gone through a removal

Table B-1 provides the list of Libby properties selected for flowerbed sampling. Samples were selected to represent soils that span a range of visible vermiculite conditions (i.e., low, moderate, and high), a range of sampling dates, and included properties both with and without the presence of other soil removal triggers. Five properties were selected for Category 1, but only four properties could be identified for Category 2. As the 2012 removal season continues, if another property can be identified that meets the selection criteria for Category 2, it will be considered for inclusion in this study.

B1.1.2 Task 2 – Flowerbeds in Troy

No new samples will be collected for Task 2. Rather, archived aliquots of previously collected flowerbed soil samples from Troy will be pulled from archive for re-analysis.

Sample selection criteria for Task 2 were developed to target soil samples collected in Troy from flowerbeds with visible vermiculite. Similar to Task 1, samples will be selected that are

representative of properties built in or prior to 1995 (Category 1) and properties built after 1995 (Category 2). In addition, because homeowner interviews conducted for Troy specifically include questions about the source of soils in flowerbeds (i.e., whether soils are store-bought), interview responses on soil source will be used to further segregate the categories as follows:

		Source of Soils in Flowerbed		
		Store-bought	Not store-bought	
property —	≤ 1995	Category 1A	Category 1B	
	> 1995	Category 2A	Category 2B	

 \leq = less than or equal to

Table B-2 provides the list of Troy flowerbed samples selected for re-analysis. A total of 12 samples were selected for re-analysis (three samples for each category condition). Samples were selected to represent soils that span a range of visible vermiculite conditions (i.e., low, moderate, and high), a range of sampling dates, and a range of PLM-VE results for LA (i.e., included both non-detect [Bin A] and trace [Bin B1] results).

B1.1.3 Task 3 – Store-bought Materials from Libby

The purpose of Task 3 is to measure potential LA concentrations in store-bought potting soil that may be placed in flowerbeds following soil removal activities. In May 2011, one sample was collected from one bag of store-bought potting soil (without vermiculite added) purchased from a local hardware store in Libby. The PLM-VE result for LA was reported as non-detect (Bin A) for this sample. In 2012, four soil samples were collected from different bags of store-bought potting soil purchased from a local hardware store in Libby. The PLM-VE results for LA were reported as non-detect (Bin A) for three samples and trace (Bin B1) for one sample. **Table B-3** summarizes all of the store-bought potting soil samples that have been collected to date and identifies the three samples that were selected for re-analysis as part of this study.

In addition, because there are no existing samples of store-bought bagged vermiculite, Task 3 will also include the collection of three vermiculite samples of bagged vermiculite that has been purchased from a local hardware store in Libby. Three different bags of vermiculite should be purchased. To the extent feasible, a range of different brands and types of vermiculite should be sampled.

B1.1.4 Task 4 – Store-bought Materials from Other Cities

The purpose of Task 4 is to measure potential LA concentrations in store-bought potting soil purchased outside of Libby to provide a frame of reference for results of potting soil purchased in Libby. The actual cities outside of Libby where these store-bought materials are purchased are not important. Thus, the selected locations are primarily dictated by ease of sample

> = greater than

collection. Because Kalispell, Montana, and Spokane, Washington are often used as flight destinations for project staff in transit to Libby, potting soil will be purchased from local hardware stores in one of these cities. In addition, because both EPA and CDM Smith have offices in Denver, Colorado, potting soil will also be purchased from local hardware stores in Denver. For each city, three different bags of potting soil (without vermiculite added) should be purchased. To the extent feasible, one of the three bags purchased should be of the same brand and type as potting soil available in Libby. The other two bags should include different brands and types of potting soil.

B1.2 Sampling Design

The following provides an overview of the two sampling efforts that will be conducted. Detailed information on sampling procedures and methods are presented in Section B2. The requirements for field QC sample collection are discussed in Section B5.1.

Sampling Flowerbeds in Libby

At each Libby property selected for sampling under (Task 1), a single 30-point composite soil sample from flowerbeds containing vermiculite will be collected. If there are multiple flowerbeds containing visible vermiculite, provided they visually appear to have similar types of soil, composite sub-sampling points should be collected from each flowerbed, with the approximate number of sub-sampling points for each flowerbed based on the relative spatial extent. Flowerbeds that appear to have distinct types of soil will be sampled separately. At least one flowerbed soil composite sample will be collected from each selected property, for a minimum of ten soil samples total. Soil from flowerpots will not be included in this investigation.

Sampling Store-bought Materials

When sampling store-bought materials (i.e., potting soil and bagged vermiculite), a single multi-point composite sample will be collected from each bag of potting soil or vermiculite. A total of nine samples will be collected from retail sources – three samples from bagged vermiculite purchased in Libby (Task 3) and six samples from potting soil purchased outside of Libby (Task 4).

B1.3 Study Variables

No two properties at the Site are exactly alike, therefore, the nature and source of soil in flowerbeds is variable. There are several potential sources of vermiculite that may be present in flowerbeds (both mine-related and non-mine-related). The categories of criteria for property selection described in Section B1.1.1 (Libby) and B1.1.2 (Troy) are designed to include a broad range of property types, such that a range of flowerbed conditions are represented.

Asbestos concentrations in soil can be heterogeneous; therefore, it is important that soil sampling methods provide an even and representative coverage of the entire flowerbed. To accomplish this, each flowerbed will be a 30-point composite sample. Each sampling point will be equally spaced such that the 30 sub-sampling points cover the entire sampling area. If the flowerbed area exceeds 1,000 square feet (ft²), the flowerbed will be split into smaller areas. Soil from multiple flowerbeds may be composited into one sample provided the total area does not exceed 1,000 ft², and the number of sub-sampling points in each flowerbed will be weighted based on their relative spatial extent.

Because it is possible that LA levels in store-bought potting soil may differ by brand and soil type, a range of brands and soil types will be evaluated.

B1.4 Critical Measurements

The critical measurement associated with this project is the measurement of the concentration of LA in flowerbed soils from residential properties in Libby, in flowerbed soils from residential properties in Troy, in store-bought potting soil and bagged vermiculite purchased in Libby, and in store-bought potting soil purchased outside of Libby. The analysis of LA could be achieved using several different analytical methods. At the Site, most soil samples are analyzed by PLM. However, PLM is not generally intended for assessing low-level (less than 1%) asbestos in soil and concentrations below 1% are not reported quantitatively. Preliminary method performance evaluations show that TEM analyses of soil prepared using the FBAS method were able to reliably quantify LA concentrations of 0.005% and lower in soil (Januch *et al.* 2012). In addition, soil concentrations are reported quantitatively (as structures per gram [s/g]). Because LA concentrations (if present) are likely to be below 1%, FBAS-TEM will be the primary preparation and analysis method for all samples collected in this study. Because PLM has been used in the past to analyze soil samples, and will be continue to be used in the future for the analysis of soil, samples will also be analyzed by PLM-VE for the purposes of comparability to other soil datasets.

B1.5 Data Reduction and Interpretation

Data collected as part of this study will be used to determine whether or not the presence of visible vermiculite in flowerbeds is a reliable indicator of mine contamination. If LA is consistently detected at elevated levels in flowerbed soils where visible vermiculite is observed, this would suggest that the presence of visible vermiculite in a flowerbed is a reliable indicator of mine contamination, and that current removal triggers are appropriate. If LA is not consistently detected (e.g., samples for Category 1 properties show detected levels of LA, but Category 2 properties do not) or is detected at levels similar to those reported in store-bought potting soils and/or borrow source and background areas in Libby, this would suggest that the presence of visible vermiculite a flowerbed is not a reliable indicator of mine contamination, and that current removal triggers may need to be re-evaluated to ensure removal activities are not being performed unnecessarily.

Data collected as part of this study will also provide information on whether other property-specific details, such as the year the property was built or if interview information on the presence of store-bought materials (as provided by the property owner), can be used to better refine the removal triggers for flowerbeds.

B2. Sampling Methods

B2.1 Sample Collection

The following subsections provide investigation-specific requirements for sample collection. A list of general field equipment that will be used to perform this sampling is provided in each of the field sampling SOPs. A medium- and investigation-specific equipment list is provided in Section B8.1 of this SAP/QAPP.

B2.1.1 Flowerbed Soil

Flowerbed soil samples will be collected, handled, and documented in general accordance with Site-specific SOP CDM-LIBBY-05, *Soil Sample Collection at Residential and Commercial Properties* (see **Appendix B**), with the following modifications:

- Flowerbeds with visible vermiculite will be sampled; soil samples will be 30-point composite samples.
- Pin flags will not be used to identify composite points within each sampling area.
- Plastic bristle brushes and aluminum foil will not be required for decontamination and storage. Instead, sampling equipment will be rinsed with locally available deionized water before and after each sample is collected.
- Soil should be collected from a depth of 0-6 inches below ground surface at each of the 30 aliquot sub-locations. Enough soil will be collected from each sub-location such that the 30-point composite yield approximately 1,000 grams of soil. Soil will be collected in a zip-top bag.
- Semi-quantitative estimation of vermiculite will performed at each aliquot sub-location as described below.

Visible Vermiculite Estimation

Visual estimation of the amount of visible vermiculite in each of the 30 sub-locations will be performed in general accordance with Site-specific SOP CDM-LIBBY-06, Semi-Quantitative Visual Estimation of Vermiculite in Soils at Residential and Commercial Properties (see **Appendix B**) with the following modifications:

- 30-point composite soil samples will be collected regardless of the presence of visible vermiculite.
- Soil will not be replaced after conducting a point inspection, but will be collected for sampling.
- Visual Vermiculite Estimation Forms will not be used. The contents of this form have been incorporated into the soil FSDS.

B2.1.2 Store-bought Materials

There is no Libby-specific SOP for sampling store-bought materials. The sampling of store-bought materials will be conducted by staff at the Sample Preparation Facility (SPF) located in Troy, MT. Thus, the CDM Smith field team is only responsible for obtaining the necessary bags of store-bought materials and providing these materials to the Troy SPF.

Store-bought potting soil will be collected as 30-point multi-increment samples, with one composite sample collected from each purchased bag of material. Samples will be 30-point composites sampled by a multi-increment approach (ITRC 2011). The entire bag contents should be placed onto a clean tarp and homogenized. Material should be spread out evenly and the 30-point composite should be collected using the 2-dimensional Japanese slab cake incremental sub-sampling technique (see Figure 6-3 of ITRC 2011 for an example), collecting about 30-35 grams of soil at each sampling point (i.e., the resulting composite sample will be approximately 1,000 grams of soil). Visual estimation of the amount of visible vermiculite (if present) at each of the 30 sub-sampling points should be recorded on the soil FSDS.

Samples of store-bought vermiculite will be collected using a grain sampler probe. Prior to inserting the probe, the bag should be rotated multiple times to ensure the bag contents are well-mixed. The probe should be inserted into the bag as many times as necessary to fill a 1-gallon zip-top bag. Following sample collection (but prior to equipment decontamination), the probe should be rinsed and the resulting rinsate collected in a wide-mouth 500-milliliter high-density polyethylene container³. The grain probe rinsate will provide information on whether asbestos fibers may have adhered to the probe walls during sampling. It is not necessary to record estimates of visible vermiculite for the vermiculite samples.

Sample handling and documentation for store-bought materials will be in general accordance with Site-specific SOP CDM-LIBBY-05, *Soil Sample Collection at Residential and Commercial Properties* (see **Appendix B**).

B2.1.3 Health & Safety Monitoring

As part of this investigation, personal air samples will also be collected during soil/vermiculite sampling activities as part of ongoing health and safety monitoring. The health and safety

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³ Headspace should be left at the top of the container to accommodate ozonation (see Section B₄).

samples will be collected using a low volume sampling pump and will be identified as 'PA-EXC' or 'PA-TWA' in the Sample Air Type field of the associated FSDS. These samples will be collected and analyzed in accordance with the *Response Action SAP* (CDM Smith 2011a).

B2.2 Global Positioning System Coordinate Collection

GPS location coordinates will be recorded in basic accordance with Site-specific SOP CDM-LIBBY-09, GPS Coordinate Collection and Handling (see **Appendix B**). For this study, GPS coordinates will only be collected during Task 1. GPS coordinates will be collected whenever the flowerbed sampling area differs from the original inspection area (i.e., whenever a new location ID will be assigned). If the original inspection area meets the criteria outlined in this SAP/QAPP, no new GPS location coordinates will be collected.

GPS coordinates will be collected as Sample Points, requiring the input of sample identification (ID) (also referred to as index ID) and location ID. Since multiple samples may be attributed to one area, for this sampling program the index ID will be input as 'N/A'.

Field-collected GPS data are converted to a usable geographic information system (GIS) format using the general processes described in SOP CDM-LIBBY-09. After the conversion from GPS points to GIS files, 100% of the data is checked visually to identify any potential data entry errors.

B2.3 Equipment Decontamination

Equipment used to collect, handle, or measure environmental samples will be decontaminated in basic accordance with Site-specific SOP EPA-LIBBY-2012-04, *Field Equipment Decontamination at Nonradioactive Sites* (see **Appendix B**). Materials used in the decontamination process will be disposed of as investigation-derived waste (IDW) as described below. This SOP specifies the minimum procedural requirements for equipment decontamination. Additional equipment decontamination procedures are also specified in the medium-specific collection SOPs.

B2.4 Handling Investigation-derived Waste

Any disposable equipment or other IDW will be handled in general conformance with Site-specific SOP EPA-LIBBY-2012-05, *Guide to Handling of Investigation-Derived Waste* (see **Appendix B**). In brief, IDW will be double bagged in clear 6-mil poly bags with 'IDW' written, in letters at least 3-inches high, in indelible ink on at least two sides of the outer bag. All IDW generated during this sampling program will remain in the custody of the sampling team until the team returns to Libby where the IDW will enter the waste stream at the local class IV asbestos landfill.

B3. Sample Handling and Custody

B3.1 Sample Identification and Documentation

B3.1.1 Sample Labels

Newly collected samples will be labeled with sample ID numbers that have been signed out by the sampling teams, as supplied by field administrative staff. Sample labels will be affixed to the inside of both the inner and outer sample bags and the sample ID number will be written in indelible ink on the outside of each bag.

Sample ID numbers will identify the samples collected during this sampling effort using the following format:

FB-####

where:

FB = Prefix that designates samples collected under this SAP/QAPP ##### = A sequential five-digit number

B3.1.2 Field Sample Data Sheets

As noted previously in Section A9, field teams will record sample information on the most current version of the Site-specific FSDS. Use of standardized forms ensures consistent documentation across samplers. Hard copy FSDSs are location-specific and allow for the entry of up to three individual samples from the same location on the same FSDS form. If columns are left incomplete due to fewer than three samples being recorded on a sheet, the blank columns will be crossed out, dated, and signed by the field team member completing the FSDS. Erroneous information recorded on a hard copy FSDS will be corrected with a single line strikeout, initial, and date. The correct information will be entered in close proximity to the erroneous entry.

FSDS information will be completed in the field before field personnel leave the sampling location. To ensure that all applicable data is accurately entered and all fields are complete, a different field team member will check each FSDS. The team member completing the hard copy form and the team member checking the form will initial the FSDS in the proper fields. In addition, the field team leader (FTL) will also complete periodic checks of FSDSs prior to relinquishment of the samples to the field sample coordinator. Once FSDSs and samples are relinquished to the field sample coordination staff, the FSDSs are again checked for accuracy and completeness when data are input into the local Scribe field database.

If a revision is required to the hard copy FSDS during any of these checks, it will be returned to the field team member initially responsible for its completion. The error will be explained to the team member and the FSDS corrected. If the team member is no longer on site, revisions will be made by sample coordination staff or the FTL. It is the responsibility of the field data manager to make the appropriate change in the local Scribe field database.

Each hard copy FSDS is assigned a unique sequential number. This number will be referenced in the field logbook entries related to samples recorded on individual sheets. Field administrative staff will manage the hard copy FSDSs in their respective field office. Original FSDSs will be filed by medium and FSDS number. Hard copies of all FSDS forms will also be sent to the CDM Smith office in Denver, Colorado for archive.

B3.1.3 Field Logbooks

The field logbook is an accounting of activities at the Site and will duly note problems or deviations from the governing documents. Field logbooks will be maintained in general conformance with Site-specific SOP EPA-LIBBY-2012-01, Field Logbook Content and Control (see **Appendix B**).

Separate field logbooks will be kept for each investigation and the cover of each field logbook will clearly indicate the name of the investigation and its sequence number. Field logbooks will be completed for each investigation activity prior to leaving a sampling location. Field logbooks will be checked for completeness and adherence to SOP requirements on a daily basis by the FTL or their designate for the first week of each investigation. When incorrect field logbook completion procedures are discovered during these checks, the errors will be discussed with the author of the entry and corrected. Erroneous information recorded in a field logbook will be corrected with a single line strikeout, initial, and date. The correct information will be entered in close proximity to the erroneous entry.

The field administrative staff will manage the field logbooks by assigning unique identification numbers to each field logbook, tracking to whom and the date each field logbook was assigned, the general investigation activities recorded in each field logbook (e.g., ambient air monitoring), and the date when the field logbook was returned. As field logbooks are completed, originals will be catalogued and maintained by the field administrative staff in their respective field office. Scanned copies of field logbooks will be maintained on the local servers for the CDM Smith offices in Libby and Denver.

B3.1.4 Photographic and Video Documentation

Photographic documentation will be collected with a digital camera in general conformance to SOP EPA-LIBBY-2012-02, *Photographic Documentation of Field Activities* (see **Appendix B**). Photographs should be taken to document representative examples of sampling locations, presampling conditions, and any other special conditions or circumstances that arise during the

sampling.

Electronic captions will be used to describe the photographs instead of maintaining photographic logs in daily logbook entries.

Photograph file names will be in the format:

Property ID_FB_date

where:

The property ID is the identifier for the property sampled or the source of the storebought materials FB indicates Flowerbed Sampling Study The date is formatted as MM-DD-YY

Digital video recordings will not be required during this investigation, unless special conditions or circumstances arise during the sampling that warrants video. File names will be in the same format as photographic documentation listed above.

B3.2 Field Sample Custody

All teams will ensure that samples, while in their possession, are maintained in a secure manner to prevent tampering, damage, or loss. All samples and FSDSs will be stored in a locked location (e.g., vehicle or hotel) at the end of each day. At the conclusion of the sampling program, the team will return to Libby and relinquish all samples and FSDSs to the sample coordinator or designated secure sample storage area. The field team will be responsible for documenting this transfer of sample custody in the logbook.

B3.3 Chain-of-Custody Requirements

The chain-of-custody (COC) is used as physical evidence of sample custody and control. This record system provides the means to identify, track, and monitor each individual sample from the point of collection through final data reporting. A complete COC record is required to accompany each shipment of samples. COC procedures will follow the requirements as stated in Site-specific SOP EPA-LIBBY-2012-06, *Sample Custody* (see **Appendix B**).

At the end of each day, all samples will be relinquished to the field sample coordinator or a designated secure storage location by the sampling team following COC procedures, and an entry will be made into the field logbook indicating the time samples were relinquished and the sample coordinator who received the samples. The field sample coordinator will follow COC procedures to ensure proper sample custody between acceptance of the sample from the field teams to delivery or shipment to the laboratory.

A member of the sample coordination staff will manually enter sample information from the hard copy FSDS into the local Scribe field project database using a series of standardized data entry forms developed in Microsoft Access by ESAT, referred to as the sample Data Entry Tool, or the "DE Tool". The DE Tool has a variety of built-in QC functions that improve accuracy of data entry and help maintain data integrity. After the data entry is checked against the hard copy FSDSs (by a different sample coordination staff member than completed the original data entry), the DE Tool is used to prepare an electronic COC. A three-page carbon copy COC will be generated from the electronic COC. The field sample coordinator will retain one hard copy of the COC for the project file; the other two hard copies of the COC will accompany the sample shipment.

The field sample coordinator will note the analytical priority level for the samples (based on consultation with the LC) at the top of the COC. A copy of the investigation-specific Analytical Requirements Summary Sheet (see **Appendix C**) will also accompany each COC.

If any errors are found on a COC after shipment, the hard copy of the COC retained by the field sample coordinator will be corrected with a single strikeout, initial, and date. A copy of the corrected COC will be provided to the LC for distribution to the appropriate laboratory. It is the responsibility of the field data manager to make any corrections to the local Scribe field project database. Sample and COC information will be published to Scribe.NET regularly from the local Scribe field project database by the field data manager (see Section B10.1 for additional details).

B3.4 Sample Packaging and Shipping

Samples will be packaged and shipped in general accordance with SOP EPA-LIBBY-2012-07, *Packaging and Shipping of Environmental Samples* (see **Appendix B**). Following sample collection, samples will be place into a sample cooler or plastic tote. Prior to sealing the container, the sample coordinator will perform a final check of the contents of the contents with the COC, sign and date the designated spaces at the bottom of the COC. The field sample coordinator will then place the custody seals on the shipping container. A custody seal will be placed over at least two sides of the sample cooler or plastic tote and then secured by tape.

The field sample coordinator will be responsible for sending samples to the appropriate location, as specified by the LC. For this study, all field-collected samples will be sent to the Troy SPF for preparation and subsequent shipment to the appropriate analytical laboratory, or archive. Samples will be hand-delivered to the Troy SPF. Samples will be packaged for transit such that they are contained and secure (i.e., will not be excessively jostled).

B3.5 Holding Times

There are no holding time requirements for the analysis of asbestos in soil or vermiculite.

B3.6 Archival and Final Disposition

All samples and grids will be maintained in storage at the Troy SPF or analytical laboratory unless otherwise directed by the EPA. When authorized by the EPA, the laboratory will be responsible for proper disposal of any remaining samples, sample containers, shipping containers, and packing materials in accordance with sound environmental practice, based on the sample analytical results. The laboratory will maintain proper records of waste disposal methods, and will have disposal company contracts on file for inspection.

B4. Analytical Methods

B4.1 Analytical Methods and Requirements

This section discusses the analytical methods and requirements for samples collected for this SAP/QAPP. This section includes detailed information on the preparation and analysis of samples, as well as the data reporting requirements, analytical turn-around times, and custody procedures.

An analytical requirements summary sheet (FLWROU4-0812), which details the specific preparation and analytical requirements associated with this sampling program, is provided in **Appendix C**. The analytical requirements summary sheet will be reviewed and approved by all participating laboratories in this sampling program prior to any sample handling. A copy of this analytical requirements summary sheet will be submitted with each COC.

B4.1.1 Sample Preparation

Newly Collected Soil Samples

All newly collected soil samples collected for asbestos analysis will be sent to the Troy SPF. Prior to preparation, all soil samples will be dried as detailed in Libby-specific SOP ISSI-LIBBY-01, *Soil Sample Preparation* (see **Appendix B**). Once dried, each sample will be split into three approximately equal portions: 1) archive aliquot; 2) FBAS-TEM aliquot; 3) PLM aliquot. The archive aliquot will be stored in accordance with SOP ISSI-LIBBY-01.

The PLM aliquot will be prepared for analysis by the Troy SPF in accordance with SOP ISSI-LIBBY-01, *Soil Sample Preparation* (see **Appendix B**). In brief, the PLM aliquot will be sieved using a ¼ inch sieve to create a fine fraction and a coarse fraction. The fine fraction will be homogenized and ground to a maximum particle size of approximately 250 micrometers (μm). This fine fraction will be further subdivided into four fractions using a riffle splitter. The coarse fraction, if any, will be sent for analysis by PLM gravimetric evaluation (i.e., PLM-Grav). One of the fine-ground fractions will be sent for analysis of LA by PLM-VE. The three remaining fine-ground fractions will be archived. The fractions submitted for analysis will be sent to an approved and accredited PLM laboratory.

The FBAS-TEM aliquot will be prepared for analysis by the FBAS laboratory in accordance with SOP ESAT-LIBBY-01, Fluidized Bed Asbestos Segregator Method for Determination of Releasable Asbestos Fibers in Soil (see **Appendix B**). In brief, the soil aliquot will be sieved using sieves with two opening sizes (6.3 millimeters [mm] and 0.85 mm). Soil material passing through the 0.85 mm sieve will be retained for use in the FBAS. For each soil sample, a total of three air filter replicates will be generated from the FBAS aliquot. Prior to generating the filter replicates, several test filters will be generated using varying amounts of soil. The particulate loading rates on these filters will be determined using phase contrast microscopy (PCM), and filter loading optimized such that the resulting filter approaches, but does not exceed, overloading. Replicate air filters for TEM analysis will then be generated using the soil mass that achieves optimum particulate loading on the filter. Replicate FBAS air filters will be sent to an approved and accredited TEM laboratory for analysis of asbestos.

Archived Soil Samples for Re-analysis

For soil samples selected for re-analysis that are currently in archive at the Troy SPF, one of the existing fine-ground fraction aliquots will be pulled from archive and sent for analysis of LA by PLM-VE. The analysis will be sent to an approved and accredited PLM laboratory, but the selected PLM laboratory should be different from the laboratory that performed the original analysis.

The original unaltered archive aliquot will also be pulled from archive and sent for preparation by the FBAS laboratory as described above.

Vermiculite Samples

Each vermiculite sample will be split into three approximately equal portions: 1) archive aliquot; 2) FBAS-TEM aliquot; 3) PLM aliquot. The archive aliquot will be stored at the Troy SPF in accordance with SOP ISSI-LIBBY-01. The PLM aliquot will be sent for analysis by NIOSH Method 9002. The FBAS-TEM aliquot will be sent for preparation by the FBAS laboratory. At the FBAS laboratory, the vermiculite sample will be sieved a 0.85 mm sieve. Any material not passing through the sieve will be lightly ground using a mortar and pestle until it is able to pass through the sieve. For each vermiculite sample, a total of three air filter replicates will be generated in accordance with SOP ESAT-LIBBY-01 (as described above). Replicate FBAS air filters will be sent to an approved and accredited TEM laboratory for analysis of asbestos.

B4.1.2 FBAS-TEM Analysis

Grid Preparation

Each replicate FBAS air filter will be used to prepare a minimum of three grids using the grid preparation techniques described in Section 9.3 of ISO 10312:1995(E). Two grids will be

examined by TEM in basic accordance with the recording procedures described in ISO 10312:1995(E), as modified by the most recent versions of Libby Laboratory Modifications LB-000016, LB-000029, LB-000066, LB-000067, and LB-000085. The remaining grid will be placed into archive.

Counting and Stopping Rules for Field Samples

To reduce the potential level of effort to complete the TEM analysis, filters will be examined using a tiered TEM magnification approach, as follows:

High Magnification Analysis

The TEM microscopist will begin the analysis utilizing a magnification of 20,000x. All amphibole structures (including not only LA but all other amphibole asbestos types as well) that have appropriate selective area electron diffraction (SAED) patterns and EDXA spectra, and having length greater than or equal to (\geq) 0.5 μ m and an aspect ratio \geq 3:1 will be recorded on the FBAS-specific TEM laboratory bench sheets and EDD spreadsheets. If observed, chrysotile structures should be recorded, but chrysotile structure counting may stop after 50 structures have been recorded.

Examine a minimum of two grid openings from each of two grids. Continue examining grid openings until one of the following is achieved:

- 1. The target analytical sensitivity $(6.3E+03 \text{ per gram } [g^{-1}])$ is achieved,
- 2. 50 LA structures are recorded, or
- 3. A total area of 1.2 mm² of filter has been examined (approximately 120 grid openings).

When one of these criteria is achieved, complete the final grid opening and stop.

Low Magnification Analysis

After completing the initial examination at 20,000x magnification, if fewer than 50 LA structures have been recorded, and the target analytical sensitivity has not yet been achieved, the TEM microscopist will switch to a lower magnification of 5,000x and continue to record only PCME LA structures (i.e., length > 5 μ m, width \geq 0.25 μ m, aspect ratio \geq 3:1) until one of the following is achieved:

- 1. The target analytical sensitivity (6.3E+03 g⁻¹) is achieved,
- 2. 50 LA structures are recorded, or
- 3. A total area of 3.0 mm² of filter has been examined (approximately 300 grid openings).

When one of these criteria is achieved, complete the final grid opening and stop.

The results for each FBAS analysis will be expressed in terms of LA structures per gram of soil or vermiculite (dry weight).

Counting and Stopping Rules for Blanks

For blanks (lot blanks, preparation blanks, and sand blanks), the TEM analyst should examine an area of 1.0 mm² (approximately 100 grid openings) utilizing a magnification of 20,000x and the counting rules described above for the "high magnification analysis".

B4.1.3 PLM Analysis

One aliquot of the fine-ground soil sample will be analyzed for asbestos using PLM-VE in accordance with Libby-specific SOP SRC-LIBBY-03, *Analysis of Asbestos Fibers in Soil by PLM* (see **Appendix B**). If there is a coarse fraction, it will be analyzed for asbestos using PLM-Grav in accordance with Libby-specific SOP SRC-LIBBY-01, *Qualitative Estimation of Asbestos in Coarse Soil by Visual Examination Using Stereomicroscopy and PLM* (see **Appendix B**). The analysis request section of the COC record will indicate the requested analyses (e.g., PLM-VE/PLM-Grav). It is the responsibility of the Troy SPF to specify the appropriate analytical method as it corresponds to the specific sample fraction being submitted for analysis (i.e., PLM-VE for fine-ground fractions or PLM-Grav for coarse fractions) on their COC records to the analytical laboratory.

Vermiculite samples will be analyzed by PLM in accordance with NIOSH Method 9002, Issue 2.

B4.1.4 Rinsate Water Analysis

Sample Preparation

The rinsate water samples (see Section B2.1.2) should be prepared for asbestos analysis in basic accordance with the techniques in EPA Method 100.2, as modified by Libby Laboratory Modification LB-000020A. In brief, the water sample will be prepared using an ozone/ultraviolet treatment that oxidizes organic matter that is present in the water or on the walls of the bottle, destroying the material that causes clumping and binding of asbestos structures. Following treatment, an aliquot of water (generally about 50 milliliters) will be filtered through a 25-millimeter diameter polycarbonate filter with a pore size of 0.1 μ m with a mixed cellulose ester filter (0.45 μ m pore size) used as a support filter.

Analysis Method

Approximately one quarter of the filter will be used to prepare a minimum of three grids using the grid preparation techniques described in Section 9.3 of ISO 10312:1995(E). Grids will be

examined by TEM in basic accordance with the recording procedures described in ISO 10312:1995(E), as modified by the most recent versions of Libby Laboratory Modifications LB-000016, LB-000029, LB-000066, LB-000067, and LB-000085.

Counting Rules

All structures with fibrous morphology, an x-ray diffraction pattern consistent with amphibole asbestos, a energy dispersive spectrum consistent with LA, length greater than or equal to $0.5\,\mu m$, and an aspect ratio (length:width) greater than or equal to $3.1\,will$ be counted and recorded. If observed, chrysotile structures will be recorded, but chrysotile structure counting may stop after $50\,structures$ have been recorded.

TEM Stopping Rules

The TEM stopping rules for equipment rinsate water samples from this investigation are specified below and were selected to be consistent with the analytical requirements specified in other water sampling efforts conducted at the Site. The stopping rules are as follows:

- 1. Count a minimum of two grid openings from each of two grids.
- 2. Continue counting until one of the following is achieved:
 - a. The target analytical sensitivity of 50,000 L-1 has been achieved.
 - b. 25 LA structures have been observed.
 - c. A total filter area of 1.0 mm² has been examined (this is approximately 100 grid openings).

When one of these criteria has been satisfied, complete the examination of the final grid opening and stop.

B4.1.5 Health and Safety Air Sample Analysis

The personal air samples collected in support of ongoing health and safety monitoring will be analyzed in accordance with the *Response Action SAP* (CDM Smith 2011). In brief, air samples will be prepared and analyzed by PCM in accordance with NIOSH Method 7400, Issue 2.

B4.2 Analytical Data Reports

An analytical data report will be prepared by the laboratory and submitted to the appropriate LC after the completion of all required analyses within a specific laboratory job (or sample delivery group). This analytical data report may vary by laboratory and analytical method but generally includes a case narrative that briefly describes the number of samples, the analyses, and any analytical difficulties or QA/QC issues associated with the submitted samples. The data report will also include copies of the signed COC forms, analytical data summaries, a QC package, and raw data. Raw data is to consist of instrument preparation logs, instrument

printouts, and QC sample results including, instrument maintenance records, COC check in and tracking, raw data instrument print outs of sample results, analysis run logs, and sample preparation logs. The laboratory will provide an electronic scanned copy of the analytical data report to the LC and others, as directed by the LC.

B4.3 Laboratory Data Reporting Tools

Standardized data reporting tools (i.e., EDDs) have been developed specifically for the Libby project to ensure consistency between different laboratories in the presentation and submittal of analytical data. In general, unique Libby-specific EDDs have been developed for each analytical method and each medium. Since the beginning of the Libby project, each EDD has undergone continued development and refinement to better accommodate current and anticipated future data needs and requirements. EDD refinement continues based on laboratory and data user input. Electronic copies of all current EDD templates are provided in the Libby Lab eRoom.

For TEM analyses, detailed raw structure data will be recorded and results will be transmitted using the Libby-specific EDDs for TEM. For PLM analyses, optical property details and results will be recorded on the Libby-specific EDDs for PLM. Standard project data reporting requirements will be met for TEM and PLM analyses. EDDs will be transmitted electronically (*via* email) to the following:

- Doug Kent, Kent.Doug@epa.gov
- Janelle Lohman, <u>Lohman.Janelle@epa.gov</u>
- Tracy Dodge, DodgeTA@cdmsmith.com
- Phyllis Haugen, HaugenPJ@cdmsmith.com
- Libby project email address for CDM Smith, <u>libby@cdmsmith.com</u>

Note: ESAT is in the process of developing a new Site-specific analytical results reporting tool, referred to as the Libby Asbestos Data Tool (LADT). This tool is a relational Microsoft® Access database with a series of standard data entry forms specific to each analytical method. The LADT creates a Microsoft® Excel export file that can be directly uploaded into an analytical Scribe project database (see Section B10.4). Laboratories have the option of using LADT as a data reporting method instead of the Libby-specific EDDs.

B4.4 Analytical Turn-around Time

Analytical turn-around time will be negotiated between the EPA laboratory coordinator (LC) and the laboratory. It is anticipated that turn-around times of 2-4 weeks are acceptable, but this may be revised as determined necessary by the EPA.

B4.5 Custody Procedures

Specific laboratory custody procedures are provided in each laboratory's *Quality Assurance Management Plan*, which have been independently reviewed at the time of laboratory procurement. While specific laboratory sample custody procedures may differ between laboratories, the basic laboratory sample custody process is described briefly below.

Upon receipt at the facility, each sample shipment will be inspected to assess the condition of the shipment and the individual samples. This inspection will include verifying sample integrity. The accompanying COC will be cross-referenced with all of the samples in the shipment. The laboratory sample coordinator will sign the COC and maintain a copy for their project files.

Depending upon the laboratory-specific tracking procedures, the laboratory sample coordinator may assign a unique laboratory identification number to each sample on the COC. This number, if assigned, will identify the sample through all further handling at the laboratory. It is the responsibility of the laboratory manager to ensure that internal logbooks and records are maintained throughout sample preparation, analysis, and data reporting.

B5. Quality Assurance/Quality Control

B5.1 Field

Field QA/QC activities include all processes and procedures that have been designed to ensure that field samples are collected and documented properly, and that any issues/deficiencies associated with field data collection or sample processing are quickly identified and rectified. The following sections describe each of the components of the field QA/QC program implemented at the Site.

B5.1.1 Training

Before performing field work in Libby, field personnel are required to read all governing field guidance documents relevant to the work being performed and attend a field planning meeting specific to the Comparative Exposure sampling effort. Additional information on field training requirements is provided in Section A8.1.

B5.1.2 *Modification Documentation*

All field deviations from and modifications to this SAP/QAPP will be recorded on the Libby field ROM form⁴. The field ROM forms will be used to document all permanent and temporary

⁴ A template of the Libby field ROM form is provided in the Libby Field eRoom (https://team.cdm.com/eRoom/R8-RAC/Libby)

changes to procedures contained in guidance documents governing investigation work that have the potential to impact data quality or usability. Any minor deviations (i.e., those that will not impact data quality or usability) will be documented in the field logbooks. ROMs are completed by the FTL overseeing the investigation/activity, or by assigned field or technical staff. As modifications to governing documents are implemented, the FTL will communicate the changes to the field teams conducting activities associated with the modification.

Each completed field ROM is assigned a unique sequential number (e.g., LFO-000026) by the CDM Smith field QAM. A ROM tracking log for all field modifications is maintained by the field QAM. This tracking log briefly describes the ROM being documented, as well as ROM author, the reviewers, and date of approval. Once a form is prepared, it is submitted to the appropriate EPA RPM for review and approval. Copies of approved ROMs are available in the Libby Field eRoom.

B5.1.3 Field Surveillances

Field surveillances consist of periodic observations made to evaluate continued adherence to investigation-specific governing documents. Because sample collection efforts will utilize field methods and procedures that are well established by seasoned field teams, it is not anticipated that a field surveillance will be performed for this investigation. However, field surveillances may be conducted if field processes are revised or other QA/QC procedures indicate potential deficiencies.

B5.1.4 Field Audits

Field audits are broader in scope than field surveillances. Audits are evaluations conducted by qualified technical or QA staff that are independent of the activities audited. Field audits can be conducted by field contractors, internal EPA staff, or EPA contracted auditors. It is the responsibility of the EPA RPM to ensure that field auditing requirements are met for each investigation. No field audits are anticipated as part of this study.

B5.1.5 Field QC Samples

Only one type of field QC sample will be collected as part of this study – field duplicates. Field duplicates are collected to help evaluate small-scale heterogeneity in the field and the precision of field sample analytical results. Field duplicates for flowerbeds will be collected from the same areas as the parent sample but from different individual sub-sampling points (i.e., a second soil sample will be collected from 30 different sub-locations within the same flowerbed). For store-bought materials, field duplicates will be collected from the same bag as the parent sample.

One field duplicate sample of soil from flowerbeds in Libby and one field duplicate of store-bought potting soil will be collected as part of this study. It is the responsibility of the FTL to ensure that the appropriate number of field duplicates is collected. Each field duplicate is given

a unique sample number, and field personnel record the sample number of the associated co-located sample in the parent sample number field of the FSDS. The same location ID is assigned to the field duplicate sample as the parent field sample. Field duplicates will be sent for analysis by the same method as field samples and are blind to the laboratories (i.e., the laboratory cannot distinguish between field samples and field duplicates).

Field duplicate results for FBAS-TEM analyses will be compared to the parent sample using the Poisson ratio test using a 90% confidence interval (Nelson 1982). The variability between the field duplicate and the associated parent field sample reflects the combined variation in sample heterogeneity and the variation due to measurement error. Because field duplicate samples are expected to have inherent variability that is random and may be either small or large, typically, there is no quantitative requirement for the agreement of field duplicates. Rather, results are used to determine the magnitude of this variability to evaluate data usability.

B5.2 Troy SPF

As noted above, prior to analysis, all collected soil and vermiculite samples will be prepared at the Troy SPF. The sections below provide detailed information on QA/QC procedures for the Troy SPF, which is maintained by adherence to standard preparation procedures, submission of preparation QC samples, facilities monitoring, and audits.

B5.2.1 Training/Certifications

Personnel performing sample preparation activities must have read and understood the *Soil Sample Preparation Work Plan*, the *SPF HASP*, and all associated SOPs and governing documents for soil preparation (e.g., SOP ISSI-LIBBY-01). In addition, all personnel must have completed 40-hour OSHA HAZWOPER training, annual updates, annual respirator fit tests, and annual or semi-annual physicals, as required.

Prior to performing activities at the Troy SPF, new personnel will be instructed by an experienced member of the SPF staff and training sessions will be documented in the SPF project files. It is the responsibility of the SPF QAM to ensure that all personnel have completed the required training requirements.

B5.2.2 *Modification Documentation*

When changes or revisions are needed to improve or document specifics about sample preparation procedures used by the Troy SPF, these changes are documented using an SPF ROM form⁵. The SPF ROM form provides a standardized format for tracking procedural

⁵ A template of the Troy SPF ROM form is provided in the Libby Lab eRoom (https://team.cdm.com/eRoom/mt/LibbyLab)

changes in sample preparation and allows project managers to assess potential impacts on the quality of the data being collected. SPF ROMs will be completed by the appropriate SPF or technical staff. Once a form is prepared, it is submitted to the ESAT QAM (or their designate) for review. Final review and approval is provided by the appropriate EPA RPM. Copies of approved SPF ROMs are available in the Libby Lab eRoom.

B5.2.3 Soil Preparation Facility Audits

Internal audits of the SPF are conducted by the SPF QAM periodically to evaluate personnel in their day-to-day activities and to ensure that all processes and procedures are performed in accordance with governing documents and SOPs. All aspects of sample preparation, as well as sample handling, custody, and shipping are evaluated. If any issues are identified, SPF personnel are notified and retrained as appropriate. Audit reports will be completed following each laboratory audit. A copy of the internal audit report, as well as any corrective action reports, will be provided to the LC and the QATS contractor.

Internal audits will be conducted following any significant procedural changes to the soil preparation processes or other SPF governing documents, to ensure the new methods are implemented and followed appropriately.

The Troy SPF is also required to participate in an annual on-site laboratory audit carried out by the EPA through the QATS contract. Audits consist of an evaluation of facility practices and procedures associated with the preparation of soil samples. A checklist of requirements, as derived from the applicable governing documents and SOPs, is prepared by the auditor prior to the audit, and used during the on-site evaluation. Evaluation of the facility is made by reviewing SPF documentation, observing sample processing, and interviewing personnel.

It is the responsibility of the QATS contractor to prepare an On-site Audit Report following the SPF audit. The On-site Audit Report includes both a summary of the audit results and completed checklist(s), as well as recommendations for corrective actions, as appropriate. Responses from each SPF to any deficiencies noted in the On-site Audit Report are also maintained with the respective reports.

It is the responsibility of the QATS contractor to prepare an On-Site Audit Trend Analysis Report on an annual basis. This report shall include a compilation and trend analysis of the on-site audit findings and recommendations. The purpose of this reported is to identify SPF performance problems and isolate the potential causes.

B5.2.4 Preparation QC Samples

Four types of preparation QC samples are collected during the soil preparation process: sand blanks, drying blanks, and preparation duplicates. Each type of preparation QC sample is described in more detail below.

Sand Blank

A sand blank is a sample of store-bought quartz sand that is analyzed to ensure that the quartz sand matrix used for drying and grinding blanks is asbestos-free. Detailed procedures for this certification process are provided in ESAT SOP PLM-02.00, *Blank Sand Certification by Polarized Light Microscopy*. In brief, about 800 grams of sand are split into 40 sand blank aliquots of roughly equal size. Each sand blank is evaluated using stereomicroscopic examination and analyzed by PLM-VE. If a sand blank has detected asbestos, it is re-analyzed by a second PLM analyst to verify the presence of asbestos. The sand is certified as asbestos-free if all 40 sand blanks are non-detect for asbestos. The sand is rejected for use if any asbestos is detected in the sand blanks. Only sand that is certified as asbestos-free will be utilized in the SPF.

Drying Blank

A drying blank consists of approximately 100 to 200 grams of asbestos-free quartz sand that is processed with each batch of field samples that are dried together (usually this is approximately 125 samples per batch). The drying blank is then processed identically to field samples. Drying blanks determine if cross-contamination between samples is occurring during sample drying. One drying blank will be processed with each drying batch per oven. It is the responsibility of the SPF QAM to ensure that the appropriate number of drying blanks is collected. Each drying blank is given unique sample number that is investigation-specific, as provided by the field sample coordinator (i.e., a subset of sample numbers for each investigation will be provided for use by the SPF). SPF personnel will record the sample number of the drying blank on the sample drying log sheet.

It is the responsibility of the QATS contractor to review the drying blank results and notify the SPF QAM immediately if drying blank results do not meet acceptance criteria and if corrective actions are necessary. If asbestos is detected in the drying blank, a qualifier of "DB" will be added to the related field sample results in the project database that were dried at the same time as the detected drying blank to denote that the associated drying blank had detected asbestos. In addition, the drying oven will be thoroughly cleaned. If asbestos continues to be detected in drying blanks after cleaning occurs, sample processing must stop and the drying method and decontamination procedures will be evaluated to rectify any cross-contamination issues.

Preparation Duplicate

Preparation duplicates are splits of field samples submitted for sample preparation. The preparation duplicates are used to evaluate the variability that arises during the soil preparation and analysis steps. After drying, but prior to sieving, a preparation duplicate is prepared by using a riffle splitter to divide the field sample (after an archive split has been created) into two approximately equal portions, creating a parent and duplicate sample.

Preparation duplicate samples are prepared at a rate of 1 per 20 samples (5%) of samples prepared. It is the responsibility of the SPF QAM to ensure that the appropriate number of preparation duplicates is prepared. Each preparation duplicate is given unique sample number that is investigation-specific, as provided by the field sample coordinator. SPF personnel will record the sample number of the preparation duplicate and its associated parent field sample on the sample preparation log sheet. Preparation duplicates are submitted blind to the laboratory for analysis by the same analytical method as the parent sample.

Preparation duplicate results will be compared to the original parent field sample using the Poisson ratio test using a 90% CI (Nelson 1982). Because preparation duplicate samples may have inherent small-scale variability that is random and may be either small or large, there is no quantitative requirement for the agreement of preparation duplicates. Rather, results are used to determine the magnitude of this variability to evaluate data usability. The QATS contractor will notify the SPF QAM when preparation duplicate results are statistically different from the parent results to determine if corrective action is needed.

B5.2.5 Performance Evaluation Standards

The USGS has prepared several Site-specific reference materials of LA in soil that are utilized as performance evaluation (PE) standards to evaluate laboratory accuracy and precision. These PE standards are kept in storage at the Troy SPF and are inserted into the sample train in accordance with SOP ISSI-LIBBY-01, with the following project-specific modification:

• PE standards will not be processed prior to insertion (i.e., no sieving or grinding of the standard should be performed).

PE standards of varying nominal levels will be inserted on a quarterly basis at a rate of at least one PE standard per analytical laboratory.

It is the responsibility of the SPF QAM to ensure that the appropriate number of PE standards is inserted. Each PE standard is given unique sample number that is investigation-specific, as provided by the field sample coordinator. SPF personnel will record the sample number of the PE standard, the nominal level of the PE standard, and whether it was inserted pre- or post-processing on the sample preparation log sheet. PE standards are submitted blind to the laboratory for analysis by the same analytical method as the field samples.

Results for PE standards will be evaluated by the QATS contractor or their designate. PE standard results that are prepared by FBAS and analyzed by TEM will be compared to results by the nominal concentration of the PE standard. The LC should be notified if PE standard results do not meet acceptance criteria. Corrective action will be taken if the PE standards demonstrate issues with accuracy and/or bias in results reporting. Examples of corrective actions that may be taken include reanalysis and/or repreparation, collaboration between and among laboratories to address potential differences in analysis methods, and analyst re-

training.

B5.3 Analytical Laboratory

Laboratory QA/QC activities include all processes and procedures that have been designed to ensure that data generated by an analytical laboratory are of high quality and that any problems in sample preparation or analysis that may occur are quickly identified and rectified. The following sections describe each of the components of the analytical laboratory QA/QC program implemented at the Site.

B5.3.1 Training/Certifications

All analytical laboratories participating in the analysis of samples for the Libby project are subject to national, local, and project-specific certifications and requirements. Additional information on laboratory training and certification requirements is provided in Section A8.2.

Laboratories handling samples collected as part of this sampling program will be provided a copy of and will adhere to the requirements of this SAP/QAPP. Samples collected under this SAP/QAPP will be analyzed in accordance with standard EPA and/or nationally-recognized analytical procedures (i.e., Good Laboratory Practices) in order to provide analytical data of known quality and consistency.

B5.3.2 Modification Documentation

All deviations from project-specific and method guidance documents will be recorded on the Libby laboratory ROM form⁶. The ROM will be used to document all permanent and temporary changes to analytical procedures. ROMs will be completed by the appropriate laboratory or technical staff. As ROMs are completed, it is the responsibility of the LC to communicate any changes to the project laboratories. When the project management team determines the need, this SAP/QAPP will be revised to incorporate necessary modifications.

Copies of approved ROMs for this SAP/QAPP will be made available in the Libby Lab eRoom.

B5.3.3 Laboratory Audits

Each laboratory working on the Libby project is required to participate in an annual on-site laboratory audit carried out by the EPA through the QATS contract. These audits are performed by EPA personnel (and their contractors), that are external to and independent of, the Libby laboratory team members. These audits ensure that each analytical laboratory meets the basic capability and quality standards associated with analytical methods for asbestos used at the

⁶ A template of the Libby laboratory ROM form is provided in the Libby Lab eRoom (https://team.cdm.com/eRoom/mt/LibbyLab)

Libby site. They also provide information on the availability of sufficient laboratory capacity to meet potential testing needs associated with the Site.

External Audits

Audits consist of several days of technical and evidentiary review of each laboratory. The technical portion of the audit involves an evaluation of laboratory practices and procedures associated with the preparation and analysis of samples for the identification of asbestos. The evidentiary portion of the audit involves an evaluation of data packages, record keeping, SOPs, and the laboratory *QA Management Plan*. A checklist of method-specific requirements for the commonly used methods for asbestos analysis is prepared by the auditor prior to the audit, and used during the on-site laboratory evaluation.

Evaluation of the capability for a laboratory to analyze a sample by a specific method is made by observing analysts performing actual sample analyses and interviewing each analyst responsible for the analyses. Observations and responses to questions concerning items on each method-specific checklist are noted. The determination as to whether the laboratory has the capability to analyze a sample by a specific method depends on how well the analysts follow the protocols detailed in the formal method, how well the analysts follow the laboratory-specific method SOPs, and how the analysts respond to method-specific questions.

Evaluation of the laboratory to be sufficient in the evidentiary aspect of the audit is made by reviewing laboratory documentation and interviewing laboratory personnel responsible for maintaining laboratory documentation. This includes personnel responsible for sample checkin, data review, QA procedures, document control, and record archiving. Certain analysts responsible for method quality control, instrument calibration, and document control are also interviewed in this aspect of the audit. Determination as to the capability to be sufficient in this aspect is made based on staff responses to questions and a review of archived data packages and QC documents.

It is the responsibility of the QATS contractor to prepare an On-site Audit Report for each analytical laboratory participating in the Libby program. These reports are handled as business confidential items. The On-site Audit Report includes both a summary of the audit results and completed checklist(s), as well as recommendations for corrective actions, as appropriate. Responses from each laboratory to any deficiencies noted in the On-site Audit Report are also maintained with the respective reports.

It is the responsibility of the QATS contractor to prepare an On-Site Audit Trend Analysis Report on an annual basis. This report shall include a compilation and trend analysis of the onsite audit findings and recommendations. The purpose of this reported is to identify common asbestos laboratory performance problems and isolate the potential causes.

Internal Audits

Each laboratory will also conduct periodic internal audits of their specific operations. Details on these internal audits are provided in the laboratory *QA Management Plan*. The laboratory QAM should immediately contact the LC and the QATS contractor if any issues are identified during internal audits that may impact data quality.

B5.3.4 Laboratory QC Analyses

General Requirements

The Libby-specific QC requirements for TEM analyses of asbestos are patterned after the requirements set forth by NVLAP. In brief, there are three types of laboratory-based QC analyses for TEM – laboratory blanks, recounts, and repreparations. Detailed information on the Libby-specific requirements for each type of TEM QC analysis, including the minimum frequency rates, selection procedures, acceptance criteria, and corrective actions are provided in the most recent version of Libby Laboratory Modification LB-000029.

With the exception of inter-laboratory analyses, it is the responsibility of the laboratory manager to ensure that the proper number of TEM QC analyses is completed. Inter-laboratory analyses for TEM will be selected *post hoc* by the QATS contractor or their designate in accordance with the selection procedures presented in LB-000029. The LC will provide the list of selected inter-laboratory analyses to the laboratory manager and will facilitate the exchange of samples between the analytical laboratories.

B6/B7. Instrument Maintenance and Calibration

B6/B7.1 Field Equipment

All field equipment (e.g., GPS units) should be maintained in basic accordance with manufacturer specifications. When a piece of equipment is found to be operating incorrectly, the piece of equipment will be labeled "out of order" and placed in a separate area from the rest of the sampling equipment. The person who identified the equipment as "out of order" will notify the FTL overseeing the investigation activities. It is the responsibility of the FLT to facilitate repair of the out-of-order equipment. This may include having appropriately trained field team members complete the repair or shipping the malfunctioning equipment to the manufacturer. Field team members will have access to basic tools required to make field acceptable repairs. This will ensure timely repair of any "out of order" equipment.

B6/B7.2 Laboratory Instruments

All laboratory instruments used for this project will be maintained and calibrated in accordance with the manufacturer's instructions. If any deficiencies in instrument function are identified, all analyses shall be halted until the deficiency is corrected. The laboratory shall maintain a log that documents all routine maintenance and calibration activities, as well as any significant repair events, including documentation that the deficiency has been corrected.

B8. Inspection/Acceptance of Supplies and Consumables

B8.1 Field

In advance of field activities, the FTL will check the field equipment/supply inventory and procure any additional equipment and supplies that are needed. The FTL will also ensure any in-house measurement and test equipment used to collect data/samples as part of this SAP/QAPP is in good, working order, and any procured equipment is acceptance tested prior to use. Any items that the FTL determines unacceptable will be removed from inventory and repaired or replaced as necessary.

The following list summarizes the general equipment and supplies required for most investigations:

- Field logbook Used to document field sampling activities and any problems in sample collection or deviations from the investigation-specific QAPPs. See Section B3.1.3 for standard procedures for field logbooks.
- Field sample data sheets (FSDSs) FSDSs are medium-specific forms that are used to document sample details (i.e., sampling location, sample number, medium, field QC type, etc.). See Section B3.1.2 for standard procedures for the completion of FSDSs.
- Sample number labels Sample numbers are sequential numbers with investigation-specific prefixes. Sample number labels are pre-printed and checked out to the field teams by the FTL or their designate. To avoid potential transcription errors in the field, multiple labels of the same sample number are prepared one label is affixed to the collected sample, one label is affixed to the hard copy FSDS form. Labels may also be affixed to the field logbook.
- Indelible ink pen, permanent marker Indelible ink pens are used to complete required manual data entry of information on the FSDS and in the field logbook (pencil may not be used). Permanent markers may also be used to write sample numbers on the sample containers.

- PPE As required by the HASP.
- Land survey map or aerial photo Used to identify appropriate sampling locations. In some cases, sketches may be added to the map/photo to designate sampling and visual inspection locations and other site features.
- Digital camera Used to document sampling locations and conditions. See Section B3.1.4 for standard procedures in photographic documentation.
- Global positioning system (GPS) unit, measuring wheel, stakes Used to identify and mark sampling locations. See B2.2 for standard procedures in GPS documentation.
- Zip-top bags Zip-top bags are used as sample containers for most types of environmental samples. Sample number labels will be affixed to the bags or the sample number will be hand-written in permanent marker on the bags.
- Decontamination equipment Used to remove any residual asbestos contamination on reusable sampling equipment between the collection of samples. See Section B2.3 for standard decontamination procedures.

In addition to the generic equipment list, the following equipment will be required for sampling activities as part of this study:

 Sampling equipment: trowel/shovel, tarp, small multi-increment scoop, grain sampler probe

B8.2 Laboratory

The laboratory manager is responsible for ensuring that all reagents and disposable equipment used in this project is free of asbestos contamination. This is demonstrated by the collection of blank samples, as described in Section B5.

B9. Non-direct Measurements

In order to identify Libby properties that match the criteria outlined in Section B1.1, the LibbyCDM_Field Scribe project database was queried (by CDM Smith) to identify properties with visible vermiculite reported in a flowerbed. The Response Manager database was queried to provide removal status information for each candidate property. Only those properties where the property investigation was performed more recently and where an outdoor soil removal is required but has not yet been performed were retained for selection in this study. The Montana Cadastral website⁷ was used to obtain county records to aide in determining the

⁷ http://svc.mt.gov/msl/mtcadastral/

year of construction for a house. If the year of construction was not available in the Montana Cadastral database, the Interior Property Inspection Forms (IPIFs) were manually reviewed in an attempt to fill any data gaps.

In order to identify Troy samples that match the criteria outlined in Section B1.1, the Troy Scribe database and Troy Owner Access Database (TOAD) was queried (by TetraTech) to identify soil samples collected from flowerbeds with visible vermiculite reported. Query output also included information on the source of the soil materials in the flowerbed (based on homeowner interview responses) and the year the property was built.

Data users will utilize the appropriate project databases to access other datasets for comparison (e.g., borrow sources and background areas). See Sections B10.4 and B10.5 for additional information on project databases and data reporting. Only those data that have undergone data verification and validation (see Section D2) and been evaluated with regard to data usability (see Section D3) should be utilized for the purposes of making comparisons.

B10. Data Management

The following subsections describe the field, Troy SPF, and analytical laboratory data management procedures and requirements for this investigation. These subsections also describe the project databases utilized to manage and report data from this investigation. Detailed information regarding data management procedures and requirements can be found in the *EPA Data Management Plan* for the Libby Asbestos Superfund Site (EPA 2012).

B10.1 Field Data Management

Scribe is a software tool developed by ERT to assist in the process of managing environmental data. A Scribe project is a Microsoft Access database. Data for the Site are captured in various Scribe projects. Additional information regarding Scribe and the Libby Scribe project databases is discussed in Section B10.3.

The field data manager utilizes a "local" field Scribe project database (i.e., LibbyCDM_Field.mdb) to maintain field sample information. The term "local" denotes that the database resides on the server or personal computer of the entity that is responsible for the creating/managing the database. It is the responsibility of the field data manager to ensure that all local field Scribe project databases are backed-up nightly to a local server.

Field sample information from the FSDS is manually entered by a member of the field sample coordination staff using a series of standardized data entry forms (i.e., DE Tool). This tool is a Microsoft Access database that was originally developed by ESAT. The DE Tool is currently maintained by CDM Smith and resides on the local server in the Libby field office. This tool is

used to prepare an electronic COC. Data in the DE Tool are imported into the local field Scribe project database by the field data manager.

It is the responsibility of the field data manager to "publish" sample and COC information from the local field Scribe database to Scribe.NET on a daily basis. It is not until a database has been published via Scribe.NET that it becomes available to external users.

B10.2 Troy SPF Data Management

The Troy SPF utilizes a local SPF Scribe project database to maintain soil sample preparation information. Soil preparation information from the preparation log sheets is entered into the local SPF Scribe project database by SPF personnel. After the data entry is checked against the original forms, it is the responsibility of the SPF manager (or their designate) to publish soil sample preparation information from the local SPF Scribe database to Scribe.NET.

B10.3 Analytical Laboratory Data Management

The analytical laboratories utilize several standardized data reporting tools developed specifically for the Libby project to ensure consistency between laboratories in the presentation and submittal of analytical data. In general, a unique Libby-specific EDD has been developed for each analytical method and each sampling medium. Electronic copies of all current EDD templates are provided in the Libby Lab eRoom.

Once the analytical laboratory has populated the EDD with results, the spreadsheet(s) are transmitted via email to the ESAT TEM Laboratory Manager, the ESAT project data manager, and the FTL (or their designate). (Other email recipients may also be specified by the ESAT LC).

The ESAT project database manager utilizes a local analytical Scribe project database (i.e., LibbyLab2012.mdb) to maintain analytical results information. The EDDs are uploaded directly into the analytical Scribe project database. It is the responsibility of the ESAT project data manager to publish analytical results information from the local analytical Scribe database to Scribe.NET.

B10.4 Libby Project Database

As noted above, Scribe is a software tool developed by ERT to assist in the process of managing environmental data. A Scribe project is a Microsoft Access database. Multiple Scribe projects can be stored and shared through Scribe.NET, which is a web-based portal that allows multiple data users controlled access to Scribe projects. Local Scribe projects are "published" to Scribe.NET by the entity responsible for managing the local Scribe project. External data users may "subscribe" to the published Scribe projects via Scribe.NET to access data. Subscription requests are managed by ERT.

All data collected for this investigation will be maintained in Scribe. As discussed above, data will be are captured in various Scribe project databases, including a field Scribe project (i.e., LibbyCDM_Field.mdb) and an analytical results Scribe project (i.e., LibbyLab2012.mdb).

B10.5 Data Reporting

Data users can access data for the Libby project through Scribe.NET. To access data, a data user must first download the Scribe application from the EPA ERT website⁸. The data user must then subscribe to each of the published Scribe projects for the Site using login and password information that are specific to each individual Scribe project. Scribe subscriptions for the Libby project are managed by ERT. Using the Scribe application, a data user may download a copy of any published Scribe project database to their local hard drive. It is the responsibility of the data user to regularly update their local copies of the Libby Scribe projects via Scribe.NET.

The Scribe application provides several standard queries that can be used to summarize and view results within an individual Scribe project. However, these standard Scribe queries cannot be used to summarize results across multiple Scribe projects (e.g., it is not possible to query both the "LibbyCDM_Field" project and the "LibbyLab2012" project using these standard Scribe queries).

If data users wish to summarize results across multiple published Scribe projects, there are two potential options. Data users may request the development of a "combined" project from ERT. This combined project compiles tables from multiple published Scribe projects into a single Scribe project. This allows data users to utilize the standard Scribe queries to summarize and view results.

Alternatively, data users may download copies of multiple published Scribe project databases for the Site and utilize Microsoft Access to create user-defined queries to extract the desired data across Scribe projects. This requires that the data user is proficient in Microsoft Access and has an intimate knowledge of proper querying methods for asbestos data for the Site.

It is the responsibility of the data users to perform a review of results generated by any data queries and standard reports to ensure that they are accurate, complete, and representative. If issues are identified by the data user, they should be reported to the EPA Region 8 data manager for resolution via email (Mosal.Jeffrey@epa.gov). It is the responsibility of the EPA Region 8 data manager to notify the appropriate entity (e.g., field, Troy SPF, analytical laboratory) in order to rectify the issue. A follow-up email will be sent to the party reporting the issue to serve as confirmation that a resolution has been reached and any necessary changes have been made.

⁸ http://www.ertsupport.org/scribe_home.htm

C ASSESSMENT AND OVERSIGHT

Assessments and oversight reports to management are necessary to ensure that procedures are followed as required and that deviations from procedures are documented. These reports also serve to keep management current on field activities.

C1. Assessment and Response Actions

C1.1 Assessments

System assessments are qualitative reviews of different aspects of project work to check the use of appropriate QC measures and the general function of the QA system. Field and office system assessments will be performed under the direction of CDM Smith's QA Director, with support from the CDM Smith QA Manager. As noted previously, it is anticipated that a field audit will be performed during this sampling program. The field audit findings will be documented in an audit report. A copy of the report will be provided to the EPA RPM and the QATS contractor. Field surveillances may be conducted if field processes are revised or other QA/QC procedures indicate potential deficiencies.

Laboratory system assessments/audits will be coordinated by the EPA. Performance assessments for the laboratories may be accomplished by submitting blind reference material (i.e., performance evaluation samples). These assessment samples are samples with known concentrations that are submitted to the laboratories without identifying them as such to the laboratories. Performance assessments will be coordinated by the EPA.

C1.2 Response Actions

Corrective response actions will be implemented on a case-by-case basis to address quality problems. Minor actions taken to immediately correct a quality problem will be documented in the applicable field or laboratory logbooks and a verbal report will be provided to the appropriate manager (e.g., the FTL or EPA LC). Major corrective actions will be approved by the EPA Remedial Project Manager and the appropriate manager prior to implementation of the change. Major response actions are those that may affect the quality or objective of the investigation. EPA project management will be notified when quality problems arise that cannot be corrected quickly through routine procedures.

In addition, when modifications to this SAP/QAPP are required, either for field or laboratory activities, a ROM must be completed by field staff and approved by the EPA prior to implementation.

C2. Reports to Management

No regularly-scheduled written reports to management are planned as part of this project. However, QA reports will be provided to management for routine audits and whenever quality problems are encountered. Field staff will note any quality problems on FSDSs or in field logbooks. Further, the CDM Smith project manager will inform EPA project management upon encountering quality issues that cannot be immediately corrected. Weekly reports and change request forms are not required for work performed under this SAP/QAPP.

D DATA VALIDATION AND USABILITY

D1. Data Review, Verification and Validation

D1.1 Data Review

Data review of Scribe project data typically occurs at the time of data reporting by the data users and includes cross-checking that sample IDs and sample dates have been reported correctly and that calculated analytical sensitivities or reported values are as expected. If discrepancies are found, the data user will contact the EPA database administrator, who will then notify the appropriate entity (field, preparation facility, or laboratory) in order to correct the issue.

D1.2 Criteria for LA Measurement Acceptability

Several factors are considered in determining the acceptability of LA measurements in samples analyzed by TEM. This includes the following:

- 1. Evenness of filter loading. This is evaluated using a chi-squared (CHISQ) test, as described in Annex F2 of ISO 10312. If a filter fails the CHISQ test for evenness, the result may not be representative of the true concentration in the sample, and the result should be given low confidence.
- 2. Results of QC samples. This includes both field and laboratory QC samples, such as field and laboratory blank samples, as well as various types of recount and re-preparation analyses. If significant LA contamination is detected in field or laboratory blanks, all samples prepared on that day should be considered to be potentially biased high. If agreement between original analyses and field or laboratory duplicates (i.e., repreparation or recount analyses) is poor, results for those samples should be given low confidence.

For PLM analyses, the following factors will be considered in determining the acceptability of LA measurements sediment samples:

- Results of PE standard analyses. PLM-VE accuracy is evaluated using LA-specific PE standards. If the results for these PE standards are not within the project-specific acceptance criteria, results should be given low confidence.
- Results of QC samples. This includes field, preparation, and laboratory QC samples. If LA contamination is detected in any blanks, associated samples should be considered to be potentially biased high. If agreement between original and repeat analyses (i.e., duplicate analyses, inter-laboratory analyses) is strongly discordant, results for those samples should be given low confidence.

D2. Verification and Validation Methods

D2.1 Data Verification

Data verification includes checking that results have been transferred correctly from the original hand-written, hard copy field and analytical laboratory documentation to the project databases. The goal of data verification is to identify and correct data reporting errors.

For analytical laboratories that utilize the Libby-specific EDD spreadsheets, data checking of reported analytical results begins with automatic QC checks that have been built into the spreadsheets. In addition to these automated checks, because these results will be reported to property owners, a detailed manual data verification effort will be performed for 100% of all samples and TEM analytical results collected as part of this sampling effort. This data verification process utilizes Site-specific SOPs (see **Appendix B**) developed to ensure TEM results and field sample information in the project databases is accurate and reliable:

- EPA-LIBBY-09 *SOP for TEM Data Review and Data Entry Verification* This Site-specific SOP describes the steps for the verification of TEM analyses, based on a review of the laboratory benchsheets, and verification of the transfer of results from the benchsheets into the project database.
- EPA-LIBBY-10 *SOP for PLM Data Review and Data Entry Verification* This Site-specific SOP describes the steps for the verification of PLM analyses, based on a review of the laboratory benchsheets, and verification of the transfer of results from the benchsheets into the project database.
- EPA-LIBBY-11 *SOP for FSDS Data Review and Data Entry Verification* This Site-specific SOP describes the steps for the verification of field sample information, based on a review of the FSDS form, and verification of the transfer of results from the FSDS forms into the project database. An FSDS review is performed on all samples selected for TEM or PLM data verification.

The data verification review ensure that any data reporting issues are identified and rectified to limit any impact on overall data quality. If issues are identified during the data verification, the frequency of these checks may be increased as appropriate.

Data verification will be performed by appropriate technical staff that are familiar with project-specific data reporting, analytical methods, and investigation requirements. The data verifier will prepare a data verification report (template reports are included in the SOPs) to summarize any issues identified and necessary corrections. A copy of this report will be provided to the appropriate project data manager, LC, and the EPA RPM. The data verifier will also transmit

the results of the data verification, including any electronic files summarizing identified discrepancies, via email to the EPA Region 8 data manager (Mosal.Jeffry@epa.gov) for resolution. A follow-up email will be sent to the data verifier to serve as confirmation that a resolution has been reached on any issues identified.

It is the responsibility of the EPA Region 8 data manager to coordinate with the FTL and/or LC to resolve any project database corrections and address any recommended field or laboratory procedural changes from the data verifier. The EPA Region 8 data manager is also responsible for electronically tracking in the project database which data have been verified, who performed the verification, and when.

D2.2 Data Validation

Unlike data verification, where the goal is to identify and correct data reporting errors, the goal of data validation is to evaluate overall data quality and to assign data qualifiers, as appropriate, to alert data users to any potential data quality issues. Data validation will be performed by the QATS contractor (or their designate), with support from technical support staff that are familiar with project-specific data reporting, analytical methods, and investigation requirements.

Data validation for asbestos should be performed in basic accordance with the draft *National Functional Guidelines (NFG) for Asbestos Data Review* (EPA 2011), and should include an assessment of the following:

- Internal and external field audit/surveillance reports
- Field ROMs
- Field QC sample results
- Internal and external laboratory audit reports
- Laboratory contamination monitoring results
- Laboratory ROMs
- Internal laboratory QC analysis results
- Inter-laboratory analysis results
- Performance evaluation results
- Instrument checks and calibration results
- Data verification results (i.e., in the event that the verification effort identifies a larger data quality issue)

A comprehensive data validation effort should be completed quarterly and results should be reported as a technical memorandum. This technical memorandum shall detail the validation procedures performed and provide a narrative on the quality assessment for each type of asbestos analysis, including the data qualifiers assigned, and the reason(s) for these qualifiers. The technical memorandum shall detail any deficiencies and required corrective actions.

The QATS contractor will also prepare an annual addendum to the *Quality Assurance and Quality Control Summary Report for the Libby Asbestos Superfund Site* (CDM Smith 2011b) to summarize results of the quarterly data validation efforts. This addendum should include a summary of any data qualifiers that are to be added to the project database to denote when results do not meet NFG guidelines and/or project-specific acceptance criteria. This addendum should also include recommendations for Site QA/QC program changes to address any data quality issues.

The data validator will transmit the results for each data validation effort via email to the EPA Region 8 data manager (Mosal.Jeffrey@epa.gov). This email should include an electronic summary of the records that have been validated, the date they were validated, any recommended data qualifiers, and their associated reason codes. It is the responsibility of the EPA Region 8 data manager to ensure that the appropriate data qualifiers and reason codes recommended by the data validator are added to the project database, and to electronically track in the project database which data have been validated, who performed the validation, and when.

In addition to performing quarterly data validation efforts, it is the responsibility of the QATS contractor (or their designate) to perform regular evaluations of all field blanks and SPF preparation blanks, to ensure that any potential contamination issues are quickly identified and resolved. If any blank contamination is noted, the QATS contractor should immediately contact the appropriate field QAM or SPF QAM to ensure that corrective actions are made.

D3. Reconciliation with User Requirements

It is the responsibility of data users to perform a data usability assessment to ensure that DQOs have been met, and reported investigation results are adequate and appropriate for their intended use. This data usability assessment should utilize results of the data verification and data validation efforts to provide information on overall data quality specific to each investigation.

The data usability assessment should evaluate results with regard to several data usability indicators. **Table D-1** summarizes several indicators of data usability and presents general evaluation methods for each indicator. Depending upon the nature of the investigation, other evaluation methods may also be appropriate. The data usability assessment results and conclusions should be included in any investigation-specific data summary reports.

Non-attainment of project requirements may result in additional sample collection or field observations in order to achieve project needs.

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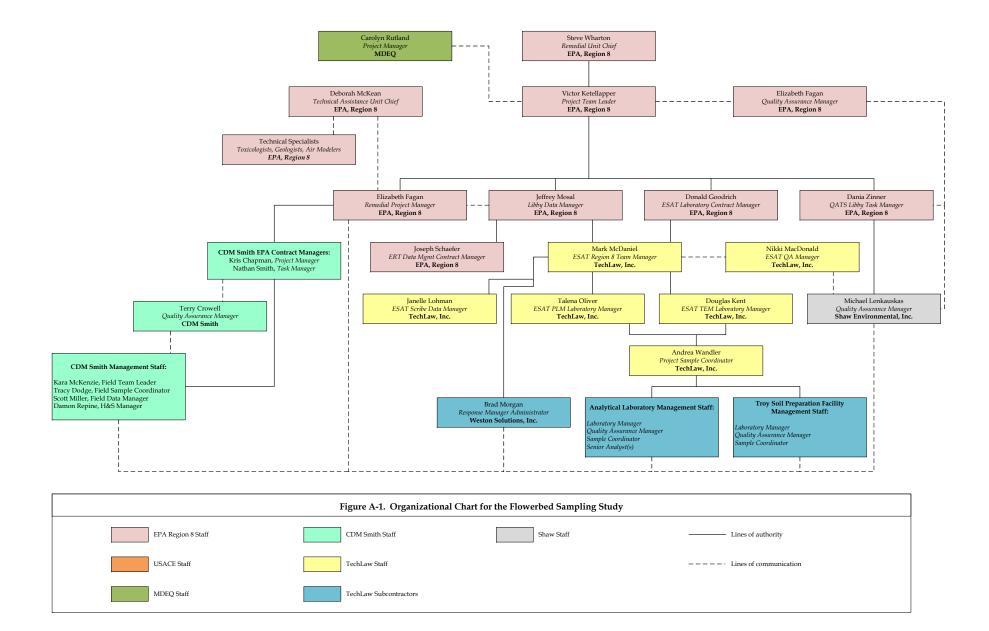
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FIGURES







TABLES



Table B-1. List of Selected Properties in Libby for Flowerbed Sampling

Category	Property ID	Address	Year Property	Location ID	VV Date	VV Sampling Point Results			
Category	Property ID	Address	Built	Location ib	v v Date	None	Low	Med	High
	AD-003339	54 Forest Way	1960	XX-001895	9/30/2011	0	2	0	0
	AD-004498	5220 Kootenai River Rd	1972	XX-009954	4/23/2012	0	0	5	0
	AD-004498			XX-010293	6/11/2012	20	19	1	0
	AD-000262	427 Reserve Rd	1978	XX-010645	5/29/2012	6	3	0	0
	AD-000202			XX-010646	5/29/2012	3	2	0	0
Category 1:	AD-004428	5186 Kootenai River Rd	1988	XX-012114	7/10/2012	2	3	0	0
Year built ≤	AD-004428			XX-010009	5/3/2012	2	3	0	0
1995	AD-002485	36 Quartz Creek Ave	1990	XX-009968	4/25/2012	3	2	0	0
				XX-010967	5/24/2012	8	2	0	0
				XX-010968	5/24/2012	5	2	0	0
				XX-010969	5/24/2012	16	5	0	0
				XX-010970	5/24/2012	7	2	0	0
				XX-010985	5/24/2012	7	1	0	0
Category 2: Year built > 1995	AD-003358	2495 Kootenai River Rd	2005	XX-011564	6/14/2012	4	1	0	0
	AD-001936	913 Sheldon Flats Rd	1996	XX-010433	5/16/2012	7	3	0	0
	AD-001930			XX-010434	5/16/2012	1	4	0	0
	AD-004113	428 Boulder Ln	1998	XX-001396	4/22/2011	0	4	6	0
	AD-005580	469 Hutton Dr	2008	XX-011050	6/4/2012	10	3	0	0
				XX-011052	6/4/2012	12	8	0	0
	AD-003300			XX-011057	6/4/2012	5	2	0	0
				XX-011062	6/4/2012	15	6	0	0

^{≤ =} less than or equal to

> = greater than

ID = identifier

VV = visible vermiculite

Table B-2. List of Selected Flowerbed Samples in Troy for Re-analysis

Catagoni		Dronorty ID	Address	Year Property Built	Flowerbed Source [1]	Sample ID	Sample Date	VV Sampling Point Results				DI MANE Deculto	
Category								Property ID	None	Low	Med	High	PLM-VE Results
Year built ≤ 1995	Store-bought materials	1A	AD-200572	209 MINERAL AVE	1971	а	TT-05176	6/16/2008	17	3	0	0	Bin A (ND)
		1A	AD-200859	818 LAKE CREEK RD	1989	а	TT-09097	7/16/2008	20	10	0	0	Bin B1 (Trace)
		1A	AD-201158	707 N 3RD ST	1930	a	TT-09252	7/21/2008	26	4	0	0	Bin A (ND)
	Not store- bought	1B	AD-200266	261 PLUGER WAY	1920	b	TT-01621	6/26/2007	28	2	0	0	Bin A (ND)
		1B	AD-200413	502A E GRANT AVE	1970	С	TT-01521	6/20/2007	2	3	0	0	Bin A (ND)
		1B	AD-201169	204 BIGHORN WAY	1986	d	TT-10724	7/8/2009	10	5	0	0	Bin B1 (Trace)
Year built > 1995	Store-bought materials	2A	AD-200255	9564 US HIGHWAY 2	2007	а	TT-02815	7/30/2007	28	2	0	0	Bin A (ND)
		2A	AD-200488	404 S 7TH ST	1997	a	TT-02686	7/24/2007	4	11	0	0	Bin B1 (Trace)
		2A	AD-201108	153 BIGHORN WAY	2004	а	TT-09118	7/17/2008	16	14	0	0	Bin A (ND)
	Not store- bought	2B	AD-201199	132 WAPITI WAY	2001	d	TT-09678	8/8/2008	10	5	0	0	Bin A (ND)
		2B	AD-200311	880 HUNTS MILL RD	2004	d	TT-12030	9/15/2010	25	5	0	0	Bin A (ND)
		2B	AD-200958	1416 US HIGHWAY 2	2000	d	TT-10375	6/16/2009	29	1	0	0	Bin A (ND)

[1] Per homeowner interview response:

- a Unexpanded/Potting soil mix, Homeowner purchase
- b Unknown
- c Expanded Unknown Source
- d Unexpanded Unknown Source

≤ = less than or equal to

> = greater than

ID = identifier

ND = non-detect

PLM-VE = polarized light microscopy, visual area estimation

VV = visible vermiculite

Table B-3. Candidate Store-bought Potting Soils for Re-analysis

Sample ID	Sample Description	Date Sampled	Laboratory	Analysis Date	PLM-VE Result	Selected for reanalysis?
1R-44826	Pallet #1 of 2, sampled 1 bag of potting soil	5/25/2011	EMSL27	5/31/2011	Bin A (ND)	Yes
FM-00025	ACE® potting soil from 2 cubic foot bag on pallet	4/26/2012	ESATR8	5/2/2012	Bin A (ND)	No
FM-00031	ACE® potting soil 2 cubic foot bag on pallet #1	5/9/2012	ESATR8	5/16/2012	Bin B1 (Trace)	Yes
FM-00032	ACE® potting soil 2 cubic foot bag on pallet #2	5/9/2012	ESATR8	5/16/2012	Bin A (ND)	No
FM-00033	ACE® potting soil 2 cubic foot bag on pallet #3	5/9/2012	ESATR8	5/16/2012	Bin A (ND)	Yes

ID = identifier

PLM-VE = polarized light microscopy, visual area estimation

ND = non-detect

EMSL = EMSL Analytical, Inc. in Libby, Montana

ESATR8 = Environmental Services Assistance Team, EPA Region 8 laboratory

Table D-1: General Evaluation Methods for Assessing Asbestos Data Usability

Data Usability Indicator	General Evaluation Method
	Sampling – Review results for co-located samples and field duplicates to provide information on variability arising from medium spatial heterogeneity and sampling and analysis methods.
Precision	Soil Preparation – Review results for preparation duplicates to provide information on variability arising from sample preparation and analysis methods.
	<u>Analysis</u> – Review results for PLM laboratory duplicates, TEM filter replicates, recounts, and repreparations to provide information on variability arising from analysis methods. Review results for inter-laboratory analyses to provide information on variability and potential bias between laboratories.
Accuracy/Bias	TEM - Calculate the background filter loading rate and use results to assign detect/non-detect in basic accordance with ASTM 6620-00. For air samples, determine the frequency of indirect preparation.
Accuracy/ bias	PLM - Review results for LA-specific soil performance evaluation standards to provide information on direction/magnitude of potential bias. Review results for blanks to provide information on potential contamination.
Representativeness	Review relevant field audit report findings and any field/laboratory ROMs for potential data quality issues.
Comparability	Compare the sample collection SOPs, preparation techniques, and analysis methods to previous investigations.
Completeness	Determine the percent of samples that were able to be successfully collected and analyzed (e.g., 99 of 100 samples, 99%).
Sensitivity	TEM - Determine the fraction of all analyses that stopped based on the area examined stopping rule (i.e., did not achieve the target sensitivity).

% = percent

ASTM = American Society of Testing and Materials

LA = Libby amphibole

QATS = Quality Assurance Technical Support ROM = record of modification

SOP = standard operating procedure

TEM = transmission electron microscopy

APPENDICES



Sampling and Analysis Plan/Quality Assurance Project Plan: Flowerbed Sampling Study Libby Asbestos Site, Operable Unit 4 Revision 0 - August 2012

Appendix A
Data Quality Objectives (DQOs)



Appendix A Data Quality Objectives for the Flowerbed Sampling Study

Data quality objectives (DQOs) are statements that define the type, quality, quantity, purpose, and use of data to be collected. The design of a study is closely tied to the DQOs, which serve as the basis for important decisions regarding key design features such as the number and location of samples to be collected and types of analyses to be performed. The U.S. Environmental Protection Agency (EPA) has developed a seven-step process for establishing DQOs to help ensure that data collected during a field sampling program will be adequate to support reliable site-specific risk management decisions (EPA 2001, 2006).

The following sections implement the seven-step DQO process associated with this study.

A.1 Step 1: State the Problem

Previous investigations conducted at residential properties at the Site have shown that soils in flowerbeds frequently contain visible vermiculite. When visible vermiculite is noted in flowerbeds during the general property investigation (GPI), soil samples are not collected from the flowerbed for asbestos analysis. This is because the presence of visible vermiculite in flowerbeds (which is considered a "specific-use area" or SUA) is a primary trigger for performing a soil removal at the property (EPA 2003). However, some property owners have indicated that, in cases where visible vermiculite is present in the flowerbed, the soils in the flowerbed were derived from store-bought potting soil, which are not expected to contain Libby amphibole (LA). This suggests that the identification of visible vermiculite in a flowerbed may not be a reliable indicator of mine contamination (i.e., LA), and that some soil removals could be performed unnecessarily as a consequence of this trigger. Therefore, the purpose of this study is to collect data that can be used to evaluate if the presence of visible vermiculite in flowerbeds is a reliable indicator of mine contamination at a property.

A.2 Step 2: Identify the Goal of the Study

The goal of this study is to collect data that can be used to determine if the presence of visible vermiculite in flowerbeds is a reliable indicator of mine contamination at a property. This study also seeks to collect data on whether other information gained about the property (e.g., year the property was built, homeowner input on flowerbed soil source) can be used to guide decision-making on soil removals for flowerbeds.

A.3 Step 3: Identify Information Inputs

The information needed to achieve the study goal consists of reliable measurements of LA concentrations in the soils from flowerbeds with visible vermiculite. Samples should be

collected from flowerbeds that span the range of soil and property conditions (e.g., year the property was built, homeowner input on flowerbed soil source) which may be present at the Site. In addition, reliable measurements of LA concentrations in store-bought materials are also needed to provide a frame of reference for the types of materials that may be present in flowerbeds at properties.

Type of Soil Sample

Soil samples should be collected using a sampling design that allows for estimation of the average level of LA in the soil (i.e., a single multi-point composite sample or multiple single-point samples from which a mean can be calculated). Results should provide an estimate of the level (e.g., mass percent [wt%], asbestos structures per gram [s/g] of soil) of LA in soil.

Analysis Method

Polarized light microscopy using visual area estimation (PLM-VE) is the typical method that is used to analyze solid media for asbestos at the Libby Site. However, PLM-VE is not generally intended for assessing low-level (less than [<] 1 percent [%]) asbestos contamination in soil. More recently, a new soil preparation method using a fluidized bed asbestos segregator (FBAS) has been utilized to allow for the analysis of soil by transmission electron microscopy (TEM). Preliminary method performance evaluations show that TEM analyses of soil prepared using the FBAS method were able to reliably quantify LA concentrations of 0.005% (on a mass basis) and lower in soil (Januch *et al.* 2012). Results from the TEM analysis provide an estimate of the LA level in soil as asbestos s/g of soil. Therefore, soils should be analyzed for asbestos by TEM after preparation using FBAS.

Because it is possible that, if present, the asbestos observed in soils from the areas of interest may be different from the type of asbestos derived from the Libby ore body at the mine site, TEM analysis results should include the size attributes (length, width) of each asbestos structure observed, along with the mineral classification (LA, other amphibole, chrysotile). Meeker *et al.* (2003) observed that most LA structures from the Libby ore body contain detectable levels of both sodium and potassium, whereas LA originating from other potential sources may not. Thus, information on the sodium and potassium content of each LA structure observed, as determined by energy dispersive spectroscopy (EDS), should also be recorded.

Because PLM has been used in the past to analyze soil samples, and will be continue to be used in the future for the analysis of soil, samples will also be analyzed by PLM-VE for the purposes of comparability to other soil datasets.

A.4 Step 4: Define the Bounds of the Study

The following sections specify the geographic (spatial) and temporal boundaries of this study.

A.4.1 Spatial Bounds

As noted above, this study seeks to collect data on LA concentrations in soil from flowerbeds with visible vermiculite. Spatial boundaries include all properties located within Operable Unit 4 (OU4; residential, commercial, and public areas of Libby) and Operable Unit 7 (OU7; the town of Troy) of the Libby Asbestos Superfund Site. The criteria for property selection are listed in Section A.7.

A.4.2 Temporal Bounds

It is not thought that the asbestos levels in soil are likely to be highly time-variable in a static environment. Thus, sample collection timing is often mainly based on ease of sample collection. However, in this particular instance, sample collection timing is driven by the need to make removal decisions before the end of the 2012 field season, as there are several properties where soil removals have been placed "on hold" pending the outcome of this study. Sampling collection should be completed as soon as possible.

A.5 Step 5: Define the Analytic Approach

Data collected as part of this study will be used to determine whether or not the presence of visible vermiculite in flowerbeds is a reliable indicator of mine contamination. Data collected as part of this study will also provide information on whether other property-specific details, such as the year the property was built or if interview information on the presence of store-bought materials (as provided by the property owner), can be used to better refine the removal triggers for flowerbeds.

Comparisons between data may be made using a variety of methods, ranging from simple visual comparisons using graphical plots to statistical comparisons using the Poisson ratio test (Nelson 1982). The Poisson ratio test can only be used in making statistical comparisons between individual samples or pooled concentrations. No statistically valid approach is available for making comparisons of asbestos datasets that cannot be pooled; therefore, these types of comparisons will rely upon graphical presentations.

A.6 Step 6: Specify Acceptance Criteria

When making statistical comparisons between two datasets, the goal is to be able to have adequate power to reject the null hypothesis if the difference between the datasets is greater than some specified level. However, because there is no statistically valid approach for making comparisons of asbestos datasets, it is not possible to calculate the number of samples required to achieve a desired statistical power. In general, more samples are needed when there is high

between-sample variability and fewer samples are needed when there is low between-sample variability. In the absence of measured data on LA in soils from flowerbeds, this study should seek to collect about 5 samples per soil condition. However, it may be nearly impossible to distinguish small differences (e.g., factor of 2-3) between datasets based on this limited sample size.

A.7 Step 7: Develop the Plan for Obtaining Data

The following sections present key elements of a sampling design that will yield data that will address the DQOs specified in Steps 1-6 above.

Property Selection Criteria - Libby

Property selection criteria should target properties in Libby that have flowerbeds with visible vermiculite. Thus, the selection criteria would be as follows:

- Property has had a detailed investigation portion of a GPI (i.e., a primary removal trigger was found in an SUA)
- Visible vermiculite was observed in a flowerbed
- Property has NOT gone through a removal

Additionally, in order to evaluate if information on the year that the property was built can be used to guide decision-making, properties should be split into two broad categories based on the year the property was built (e.g., pre/post 1995). As noted above, ideally, about five properties should be selected for each category for flowerbed soil sample collection.

Property Selection Criteria - Troy

Unlike Libby, because the Troy sampling program has collected soils from flowerbeds with visible vermiculite, no new samples need to be collected. Rather, archived aliquots of previously collected flowerbed soil samples from Troy can be re-analyzed to support this study.

Sample selection criteria are similar to those for Libby. Samples should be representative of soils collected from flowerbeds in Troy with visible vermiculite. In order to evaluate if information on the year that the property was built can be used to guide decision-making, properties should be split into two broad categories based on the year the property was built (e.g., pre/post 1995). In addition, because homeowner interviews conducted for Troy specifically include questions about the source of soils in flowerbeds (i.e., whether soils are store-bought), interview responses on soil source should also be used to further split these categories. About five properties should be selected for each category.

TEM Analytical Requirements

As noted above, soil samples should be prepared by FBAS and resulting filters should be analyzed by TEM in accordance with ISO 10312:1995(E) counting and recording rules (as modified by the Libby-specific laboratory modifications).

In general, three alternative stopping rules are specified to ensure data collected by TEM analysis are adequate:

- 1. The target analytical sensitivity (TAS) to be achieved
- 2. A maximum number of asbestos structures to be counted
- 3. A maximum area of filter to be examined

The basis for each of these values for this study is presented below.

Target Analytical Sensitivity

At present, there is no risk-based cleanup level for asbestos in soil. Therefore, there is no firm basis for selecting the TAS. Based on the available PLM-VE results for flowerbed soil samples from Troy, which tended to be Bin A (non-detect) and Bin B1 (trace), it is likely that concentrations will be low (<0.2%). Previous soil studies where FBAS-TEM analyses of borrow source and back ground areas have been conducted have utilized a TAS of 6.3E+03 (g)-1. This level of analytical sensitivity should be sufficient to allow reliable quantitation of soil samples containing asbestos levels of 0.001% by mass (about 1E+04 s/g). This TAS should suffice to provide adequate quantitative results for this study and maintain comparability to other soil studies conducted at the Site.

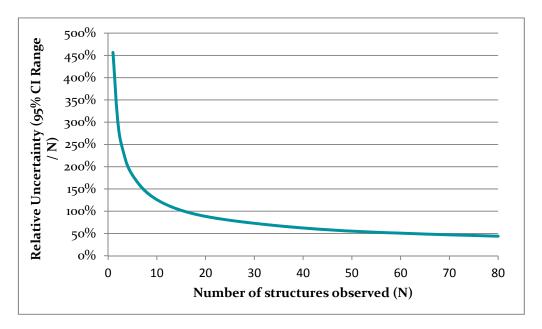
Maximum Number of Asbestos Structures

Ideally, all samples would be examined by TEM until the TAS is achieved. However, for filters that have high asbestos loading, reliable estimates of soil concentration may be achieved before achieving the target analytical sensitivity. This is because the uncertainty around a TEM estimate of asbestos concentration in a sample is a function of the number of structures observed during the analysis. The 95% confidence interval (CI) around a count of N structures is computed as follows:

```
Lower bound (2.5%) = \frac{1}{2} · CHIINV(0.975, 2 · N<sub>observed</sub> + 1)
Upper bound (97.5%) = \frac{1}{2} · CHIINV(0.025, 2 · N<sub>observed</sub> + 1)
```

As $N_{observed}$ increases, the absolute width of the CI range increases, but the relative uncertainty (expressed as the CI range divided by $N_{observed}$) decreases. This concept is illustrated the figure below.

Relationship Between Number of Structures Observed and Relative Uncertainty



The goal is to specify a target N such that the resulting Poisson variability is not a substantial factor in the evaluation of method precision. A preliminary evaluation of the performance evaluation (PE) standard results show that Poisson counting variation was an important contributor to the observed between-replicate variability (especially when fewer than 50 structures were observed). However, above about 50 structures, there is little change in the relative uncertainty. Therefore, the count-based stopping rule for TEM should utilize a maximum structure count of about 50 structures.

Maximum Area to be Examined

The area of filter that must be examined to achieve the target analytical sensitivity is calculated as:

Area (mm²) = EFA / (TAS · f · M ·
$$Q_R$$
)

where:

EFA = Effective filter area (mm²)

 Q_R = Flow ratio^a

TAS = Target analytical sensitivity (g)-1

f = fraction of filter used in the TEM examination

M = Mass of soil (assumed to be 2.0 g)

^a The ratio of the air volume that is captured on the filter to the total air volume passed through the sample. Q_R is calculated by dividing the air flow rate through the air filter cassette by the total air flow rate through the sample: $Q_R = 0.2 \text{ L/min} / 16 \text{ L/min} = 0.0125$

Based on a target analytical sensitivity of 6.3E+03 g⁻¹, the filter area that would need to be examined (assuming a direct preparation, f = 1) to achieve the target analytical sensitivity is:

Area =
$$855 / (6.3E+03 \cdot 1 \cdot 2 \cdot 0.125) = 0.55 \text{ mm}^2$$

Assuming that each grid opening has an area of about 0.01 mm², this would correspond to about 246 grid openings.

In order to limit the maximum effort expended on any one sample and to accommodate project budget constraints, a maximum examination area of 3.0 mm² is identified for this project. Assuming that each grid opening has an area of about 0.01 mm², this would correspond to about 300 grid openings.

Summary of Stopping Rules:

Based on the discussions above, the stopping rules for this project should be as follows:

- 1. Examine a minimum of 2 grid openings from each of two grids.
- 2. Continue examining grid openings until one of the following is achieved:
 - a. The target sensitivity of 6.3E+03 g⁻¹ is achieved.
 - b. 50 total asbestos structures have been recorded.
 - c. A total filter area of 3.0 mm² has been examined (this is approximately 300 grid openings).

When one of these criteria has been satisfied, complete the examination of the final grid opening and stop.

A.7.6 Refining the Study Design

In accordance with the EPA's DQO process, it is expected that the sampling program described in this document may be modified as data are obtained. For example, the analytical requirements may be refined depending on the detection frequency, mean values, and sample variability observed in the sample results.

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Sampling and Analysis Plan/Quality Assurance Project Plan: Flowerbed Sampling Study Libby Asbestos Site, Operable Unit 4

Revision 0 - August 2012

Appendix B Standard Operating Procedures (SOPs)

SOP ID	SOP Description				
Field Procedures					
EPA-LIBBY-2012-01	Field Logbook Content and Control				
EPA-LIBBY-2012-02	Photographic Documentation of Field Activities				
EPA-LIBBY-2012-04	Field Equipment Decontamination				
EPA-LIBBY-2012-05	Handling Investigation-Derived Waste				
EPA-LIBBY-2012-06	Sample Custody				
EPA-LIBBY-2012-07	Packaging and Shipping of Environmental Samples				
CDM-LIBBY-05	Site-Specific SOP for Soil Sample Collection				
CDM-LIBBY-06	Semi-Quantitative Visual Estimation of Vermiculite in Soils at				
CDW-LIDD1-00	Residential and Commercial Properties				
CDM-LIBBY-09	GPS Coordinate Collection and Handling				
Laboratory Procedures					
EPA-LIBBY-08	Indirect Preparation of Air and Dust Samples for Analysis by TEM				
ISSI-LIBBY-01	Soil Sample Preparation				
ESAT-LIBBY-01	Fluidized Bed Asbestos Segregator Method for Determination of				
ESAT-LIBDT-01	Releasable Asbestos Fibers in Soil				
ESAT SOP PLM-02.00 Blank Sand Certification by Polarized Light Microsco					
Data Verification Procedures					
EPA-LIBBY-09	TEM Data Review and Data Entry Verification				
EPA-LIBBY-11	FSDS Data Review and Data Entry Verification				

The most recent versions of all field SOPs are provided electronically in the Libby Field eRoom (https://team.cdm.com/eRoom/R8-RAC/Libby).

The most recent version of all laboratory and data verification SOPs are provided electronically in the Libby Lab eRoom (https://team.cdm.com/eRoom/mt/LibbyLab).



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Appendix C Analytical Requirements Summary Sheet [FLWROU4-0812]

The most recent version of the Analytical Requirements Summary Sheet is provided electronically in the Libby Lab eRoom (https://team.cdm.com/eRoom/mt/LibbyLab).



Requirements Summary: #FLWROU4-0812 Requirements Revision #: 0

Effective Date: August 14, 2012

SAP/QAPP REQUIREMENTS SUMMARY #<u>FLWROU4-0812</u> SUMMARY OF PREPARATION AND ANALYTICAL REQUIREMENTS FOR ASBESTOS

Title: Sampling and Analysis Plan/Quality Assurance Project Plan, Flowerbed Sampling Study, Operable Unit 4, Libby Asbestos Site

SAP Date (Revision): August 2012 (Revision 0)

EPA Technical Advisor: Elizabeth Fagen (303-312-6095, Fagen. Elizabeth@epa.gov) (contact to advise on DQOs of SAP related to preparation/analytical requirements)

Sampling Program Overview: This program consists of four tasks: 1) sampling and analysis of flowerbed soils in Libby, 2) re-analysis of flowerbed soils from Troy, 3) sampling and analysis (or re-analysis) of store-bought materials from Libby, and 4) sampling and analysis of store-bought materials from other cities. As part of this program, soil samples will be prepared by fluidized bed asbestos segregator and analyzed for asbestos by TEM. Samples will also be analyzed by PLM-VE. Personal air samples will also be collected for H&S monitoring and analyzed by PCM.

Sample ID Prefix: FB-____

Estimated number and timing of field samples:

All samples will be collected/analyzed in August 2012 timeframe (exact dates have not yet been determined). Estimated numbers of samples below do not include field QC.

- >> Libby, flowerbed soil = 10 samples
- >> Troy, flowerbed soil = 12 samples
- >> Libby, store-bought = 6 samples (3 soil, 3 vermiculite)
- >> Other cities, store-bought soil = 6 samples
- >> Grain probe rinsates = 3 samples

Effective Date: August 14, 2012

1. H&S PERSONAL AIR

PCM Preparation and Analytical Requirements for Air Field Samples:

			Preparati	on Details			Anal	ysis Details	Amplicable Laboratory
Medium	Medium,	Investi-	Indirect	Prep? (a)	Filter		Recording	Analytical Sensitivity/Prioritized	Applicable Laboratory Modifications
Code	Sample Type	gative?	With	Without	Archive?	Method	Rules	Stopping Rules	(current version of)
		gative	Ashing	Ashing	Archive:		Ruics	Stopping Rules	(current version or)
A	Air, Health &	No	No	Yes, if	Yes	PCM –	For PCM:	For PCM: Count a minimum of	For PCM: LB-000015
	Safety			material is		NIOSH	NIOSH 7400,	20 FOVs, then continue counting	
				overloaded		7400,	"A" rules	until one is achieved:	For AHERA:
				(>25%) or		Issue 2		i) 100 fibers are recorded	LB-000029, LB-000031,
				unevenly			If AHERA is	ii) 100 FOVs are examined	LB-000067, LB-000085
				loaded on		TEM-	<u>requested:</u>	(regardless of count)	
				filter		AHERA	All asbestos;		
						(upon	$L \ge 0.5 \mu m$	For AHERA: Examine 0.1 mm ²	
						request)	$AR \ge 5:1$	of filter	

⁽a) See most current version of SOP EPA-LIBBY-08 for preparation details.

PCM Preparation and Analytical Requirements for Air Field Quality Control Samples:

	Medium,	Pre	eparation De	etails		Analysi	is Details	Applicable Laboratory
Medium	Sample	Indired	et Prep?			Recording		Applicable Laboratory Modifications
Code	Type	With	Without	Archive?	Method	Rules	Stopping Rules	(current version of)
	Type	Ashing	Ashing			Kules		(current version or)
В	Air, Health	No	No	Yes	PCM – NIOSH	For PCM:	For PCM: Count a minimum of 20	For PCM: LB-000015
	& Safety,				7400, Issue 2	NIOSH 7400,	FOVs, then continue counting until	
	field blank					"A" rules	one is achieved:	For AHERA:
					TEM-AHERA		i) 100 fibers are recorded	LB-000029, LB-000031,
					(upon request)	If AHERA is	ii) 100 FOVs are examined	LB-000067, LB-000085
						requested:	(regardless of count)	
						All asbestos;		
						$L \ge 0.5 \mu m$	For AHERA: Examine 0.1 mm ² of	
						$AR \ge 5:1$	filter	

Requirements Revision #: 0 Effective Date: August 14, 2012

2. SOIL AND VERMICULITE

Soil Preparation and Analysis Requirements:

Preparation Method	Analysis Method (b)	Applicable Laboratory Modifications (current version of)
Soil PLM: ISSI-LIBBY-01, Rev. 11 Fluidized Bed: ESAT-LIBBY-01, Rev. 0 Vermiculite PLM: None Fluidized Bed: ESAT-LIBBY-01, Rev. 0 (with light grind with mortar and pestle, if needed)	Soil PLM-Grav: SRC-LIBBY-01, Rev. 3 PLM-VE: SRC-LIBBY-03, Rev.3 TEM: Modified ISO (see below) Vermiculite PLM: NIOSH 9002 TEM: Modified ISO (see below)	PLM: none TEM: see below

⁽b) For PLM-VE re-analyses, the PLM-VE analysis should be performed by a different laboratory than who performed the original analysis.

TEM Analysis Requirements for Samples Prepared by Fluidized Bed:

			Preparati	on Details			Analy	sis Details	Applicable Laboratory
Medium Code	Medium, Sample Type	Investi- gative?	Indirect P With Ashing	rep? (c,d) Without Ashing	Filter Archive?	Method	Recording Rules (e)	Analytical Sensitivity/Prioritized Stopping Rules (f,g)	Modifications (current version of)
С	FBAS Filter	Yes	Yes	No	Yes	TEM – Modified ISO 10312	$\frac{\text{High Mag}}{(20,000x, \text{Initial}):}$ $All \ asbestos$ $L: \geq 0.5 \ \mu\text{m}$ $AR: \geq 3:1$ $\frac{\text{Low Mag}}{(5,000x,}$ $\frac{\text{Supplemental}):}{\text{All asbestos;}}$ $L: > 5 \ \mu\text{m}$ $W: \geq 0.25 \ \mu\text{m}$ $AR: \geq 3:1$	High Mag: Count a minimum of 2 grid openings in 2 grids, then continue counting until one is achieved: i) sensitivity of 6.3E+03 g ⁻¹ is achieved ii) 50 LA structures are recorded iii) 1.2 mm ² of filter has been examined Low Mag: Count until one is achieved: i) sensitivity of 6.3E+03 g ⁻¹ is achieved ii) 50 LA structures are recorded (including the LA structures counted at high mag) iii) 3.0 mm ² of filter has been	LB-000016, LB-000029, LB-000066, LB-000067, LB-000085
								examined (including the filter area counted at high mag)	

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- (c) The filter analyzed in the TEM must be from 10 to 30% loaded without uneven loading. If this is not achieved, contact the FBAS preparation laboratory to request a new FBAS filter submittal. Laboratories may elect to not analyze a filter that is 25% to 30% loaded if too many overlapping particles are observed based on professional judgment and request a new filter submittal. If a sample is suitable for direct preparation (properly loaded with no loose debris) then it should be prepared directly. If a sample is not suitable for direct preparation, then it should undergo rock flour preparation in accordance with SOP EPA-LIBBY-08.
- (d) A total of 3 replicate FBAS filters will be generated for each soil sample.
- (e) If observed, chrysotile structures should be recorded, but chrysotile structure counting may stop after 50 structures have been recorded.
- (f) Only proceed with low magnification analysis if the high magnification analysis recorded fewer than 50 LA structures and the target analytical sensitivity was not achieved.

(g) Structure morphology photos are required for the first 5 LA structures found per sample.

TEM Analysis Requirements for Fluidized Bed Preparation Quality Control Samples:

	Medium Sample Type		Preparation Details			Analysis Deta	Applicable Laboratory		
Medium			Indirect Prep?			Recording		Applicable Laboratory Modifications	
Code	Sample Type	With	With Without Archive? Ashing Ashing		Method Rules		Stopping Rules	(current versions of)	
		Ashing	Ashing						
D	Preparation Blank, Lot Blank, Sieve Blank	No	No	Yes	TEM – ISO 10312 (High Mag, 20,000X)	All asbestos; $L \ge 0.5 \mu m$ $AR \ge 3:1$	Examine 1.0 mm ² of filter area	LB-000016, LB-000029, LB-000066, LB-000067, LB-000085	

3. EQUIPMENT RINSATE WATER

TEM Preparation and Analytical Requirements for Water Samples:

		·	Preparat	tion Details (h)			Anal	ysis Details	Applicable Laboratory
Medium Code	Medium	Investi- gative?	Indir With Ashing	ect Prep? Without Ashing	Filter Archive?	Method	Recording Rules (i)	Analytical Sensitivity/ Stopping Rules	Modifications (current version of)
Е	Rinsate Water	Yes	No	No	Yes	Standard TEM; ISO 10312	All asbestos; $L: \geq 0.5 \mu m$ $AR: \geq 3:1$	Count a minimum of 2 grid openings in 2 grids, then continue counting until one is achieved: i) sensitivity of 50,000 L ⁻¹ is achieved ii) 25 structures are recorded iii) A total filter area of 1.0 mm ² has been examined (approx. 100 grid openings)	LB-000016, LB-000029, LB-000066, LB-000067, LB-000085

⁽h) Sample and filter preparation should be performed in basic accordance with EPA Method 100.2 (as modified by LB-000020A). Grid preparation should be performed in basic accordance with Section 9.3 of ISO 10312:1995(E).

⁽i) If observed, chrysotile structures should be recorded, but chrysotile structure counting may stop after 50 structures have been recorded.

Requirements Summary: #FLWROU4-0812

Requirements Revision #: 0 Effective Date: August 14, 2012

Analytical Lab	oratory Quality	V Control Sam	ple Frequencies

<u>TEM (j):</u> Lab Blank – 4%

<u>PLM (k):</u>

Lab Duplicate Cross-Check- 8% Lab Duplicate Self-Check- 2% PCM (1): Blind Recounts – 10%

Recount Same – 1% Recount Different – 2.5%

> Verified Analysis – 1% Interlab – 0.5%

Interlab – 0.5% Repreparation – 1%

- (j) See LB-000029 for selection procedure and QC acceptance criteria
- (k) See SRC-LIBBY-03 for QC acceptance criteria
- (1) See NIOSH 7400 for QC acceptance criteria

Requirements Revision:

0 8/14/2012 N/A	Revision #:	Effective Date:	Revision Description
v v v v v v v v v v v v v v v v v v v	0	8/14/2012	N/A

Analytical Laboratory Review Sign-off:

⊠ EMSL – Libby [sign & date: R.K. Mahoney 15 August 2012]	⊠ ESAT [sign & date: _Douglas_Kent_14_August_2012
⊠ EMSL – Cinnaminson [sign & date: R. Denton 16 August 2012]	☐ Hygeia [sign & date: _Kyeong Corbin 15 August 2012 _
⊠ EMSL – Beltsville [sign & date: _Joseph Centifonti 15 Aug. 2012]	□ RESI [sign & date:Jeanne Spencer 8/15/2012]
⊠ EMSL – Denver [sign & date: E. Orthun 8.16.12]	

[Checking the box and initialing above indicates that the laboratory has reviewed and acknowledged the preparation and analytical requirements associated with the specified SAP.]